U.S. DEPARTMENT OF COMMERCE
National Technical Information Service

PB-280 495

Field and Laboratory Methods Applicable to Overburdens and Minesoil

West Virginia Univ, Morgantown Coll of Agriculture and Forestry

Prepared for

Industrial Environmental Research Lab, Cincinnati, Ohio

Mar 78

PB 280 495

EPA-600/2-78-054
March 1978

Environmental Protection Technology Series

FIELD AND LABORATORY METHODS APPLICABLE TO OVERBURDENS AND MINESOILS

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TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO.
EPA-600/2-78-054

2.

3. RECIPIENT'S ACCESSION NO.
PB280495

4. TITLE AND SUBTITLE
FIELD AND LABORATORY METHODS APPLICABLE TO OVERBURDENS AND MINESOILS

5. REPORT DATE
March 1978 issuing date

6. PERFORMING ORGANIZATION CODE

7. AUTHOR(S)
Andrew A. Sobek, William A. Schuller, John R. Freeman, and Richard M. Smith

8. PERFORMING ORGANIZATION REPORT NO.

9. PERFORMING ORGANIZATION NAME AND ADDRESS
College of Agriculture and Forestry
West Virginia University
Morgantown, West Virginia 26506

10. PROGRAM ELEMENT NO.
EHB - 526

11. CONTRACT/GRANT NO.
R 803508-01-0

12. SPONSORING AGENCY NAME AND ADDRESS
Industrial Environmental Research Laboratory - Cin., OH
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, Ohio 45268

13. TYPE OF REPORT AND PERIOD COVERED
Final 1/75-12/76

14. SPONSORING AGENCY CODE
EPA/600/12

15. SUPPLEMENTARY NOTES

16. ABSTRACT

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Incorporated within this manual are step-by-step procedures on field identification of common rocks and minerals; field sampling techniques; processing of rock and soil samples; and chemical, mineralogical, microbiological, and physical analyses of the samples. The methods can be used by mining companies, consultant firms, and State and Federal agencies to insure mining efficiency, post-mining land and water quality, and long range land use.

Inherent to these methods is the definition of terms. Many common terms are used inconsistently even within small groups; and when multiple disciplines are involved, communication demands that many terms must be defined for that particular purpose. Thus, the definition of essential rock, soil, chemical, mineralogical, microbiological, and physical terms constitute an important part of this project.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
*Coal Mines, *Surface Mining, *Overburden, *Chemical Properties *Weathering	*Potential Toxicity, *Minesoils, *Neutralization Potentials, *Available Nutrients, *Physical Properties, *Acid-Base Account, *Preplanning, *Analytical Properties, *Mineralogical Properties, *Microbiological Properties	43F 48A 91A

18. DISTRIBUTION STATEMENT Release to Public	**19. SECURITY CLASS** *(This Report)* Unclassified	**21. NO. OF PAGES** 219
	20. SECURITY CLASS *(This page)* Unclassified	**22. PRICE** A10 - A01

EPA Form 2220-1 (9-73)

☆ U. S. GOVERNMENT PRINTING OFFICE: 1978-759-829

EPA-600/2-78-054
March 1978

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Grant No. R803508-01-0

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FOREWORD

When energy and material resources are extracted, processed, converted and used, the related pollutional impacts on our environment and even our health often require that new and increasingly more efficient pollution control methods are used. The Industrial Environmental Research Laboratory - Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet those needs both efficiently and economically.

This report provides chemical, physical, mineralogical, and microbiological procedures for the analysis of coal overburdens and the resultant minesoils. These step-by-step methods identify and measure rock and soil properties that influence advance planning, mining efficiency, post-mining land and water quality and long range land use.

Rock and soil property measurements will be especially useful to State and Federal agencies, private contractors, and mining firms who require detailed information for pre-mining planning and projections of future results expected under specified management. For further information contact the Extraction Technology Branch of the Resource Extraction and Handling Division.

David G. Stephan
Director
Industrial Environmental Research Laboratory
Cincinnati

ABSTRACT

With the growing demand for environmental assessment of a mining site, it becomes apparent that a manual of field and laboratory procedures to study the overburden and the resulting minesoil is necessary.

Incorporated within this manual are step-by-step procedures on field identification of common rocks and minerals; field sampling techniques; processing of rock and soil samples; and chemical, mineralogical, microbiological, and physical analyses of the samples. The methods can be used by mining companies, consultant firms, and State and Federal agencies to insure mining efficiency, post-mining land and water quality and long range land use.

Inherent to these methods is the definition of terms. Many common terms are used inconsistently even within small groups; and when multiple disciplines are involved, communication demands that many terms must be defined for that particular purpose. Thus, the definition of essential rock, soil, chemical, mineralogical, microbiological, and physical terms constitute an important part of this project.

This report was submitted in fulfillment of Project R803508-01-0 by West Virginia University under the sponsorship of the U.S. Environmental Protection Agency. Work was completed as of December, 1976.

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ABBREVIATIONS

a -- acre(s)
cc -- cubic centimeter(s)
CEC -- Cation Exchange Capacity
cm -- centimeter(s)
ft -- foot (feet)
g -- gram(s)
in -- inch(es)
kg -- kilogram(s)
km -- kilometer(s)
l -- liter(s)
lb -- pound(s)
m -- meter(s)
M -- Molar
meq -- milliequivalent(s)
meq/100g -- milliequivalents per 100 grams
mg -- milligram(s)
min -- minute(s)
ml -- milliliter(s)
mm -- millimeter(s)
mmhos/cm -- millimhos per centimeter
MPN -- Most Probable Numbers
N -- Normal
nm -- nanometer(s)
oz -- ounce(s)
ppm -- parts per million
pp2m -- parts per 2 million
% -- percent
psi -- pounds per square inch
RPM -- revolutions per minute
S.M.P. -- Shoemaker, McLean, and Pratt
t -- ton(s)

ACKNOWLEDGMENTS

The following organizations have assisted in this work: West Virginia Steering Committee for Surface Mine Research; West Virginia Surface Mine and Reclamation Association; West Virginia Department of Natural Resources; West Virginia Geological and Economic Survey; United States Department of Agriculture, Soil Conservation Service; and a number of mining companies whose employees provided active cooperation that made this study possible.

Valuable consultation and advice were provided by: Alan C. Donaldson, Frank W. Glover, Walter E. Grube, Jr., Everett M. Jencks, John J. Renton, John C. Sencindiver, and Rabindar N. Singh.

The following people participated in the preparation of this report: Thomas Arkle, Jr., Carlos P. Cole, John R. Freeman, Milton T. Heald, Eric F. Perry, William A. Schuller, Richard Meriwether Smith, Andrew A. Sobek, Ronnie L. Taylor, and Harold A. Wilson.

Assistants in the field and laboratory work included: Sammy L. Baldwin, Shelia A. McFarland, Robert A. Philips, and Alfred N. Wickline.

For the reader who may have need to confer with the authors of individual major topics, the following list is provided:

Introduction, Units, Conversions, and Preplanning (Arkle, Smith, Sobek).

What to Look For and Measure in the Field (Freeman, Perry, Schuller, Smith, Sobek, Taylor).

Overburden Sampling and Labeling (Freeman, Perry, Schuller, Smith, Sobek).

Minesoils (Freeman, Sencindiver, Smith, Sobek).

Characterizing, Subsampling, and Crushing Samples (Freeman, Perry, Schuller, Sobek).

Chemical Methods (Freeman, Schuller, Smith, Sobek).

Mineralogical Methods (Heald, Schuller).

Physical Methods (Perry, Schuller, Sobek).

Microbiological Methods (Schuller, Smith, Wilson).

Short Term and Simulated Weathering (Freeman, Schuller, Smith, Sobek, Taylor).

The support of the project by the Office of Research and Development, U.S. Environmental Protection Agency, and the help provided by Robert B. Scott in finalizing this report; Elmore C. Grim, Grant Project Officer; and Ronald D. Hill is gratefully acknowledged.

SECTION 1

INTRODUCTION, UNITS, CONVERSIONS, AND PREPLANNING

1.1 INTRODUCTION

Studies of coal overburdens and minesoils in relation to environmental quality have progressed to the point that needed appraisals can be made with confidence if adapted and calibrated procedures are followed in the field and the laboratory. This manual contains such procedures. They are described in a step-by-step manner that should assure consistency of results. These procedures include everything from identification of common rocks and minerals in the field through interpretation of analytical results.

When a manual is to be used by many different disciplines, many terms must be defined for a particular purpose or misunderstandings result. Insofar as the application of this manual is concerned, essential rock, soil, chemical, physical, and engineering terms have been defined. Outside of the manual, other meanings may be attached to these terms.

This manual consists of four major sections: Section 1 introduces the manual and contains the advance planning approach; Section 2 contains all procedures and clues to be used in the field; Section 3 is strictly laboratory methods; and Section 4 is a combination of both field and laboratory weathering methods.

Each section is subdivided into specific groupings of closely related material. In turn, these subsections are subdivided into individual procedures. They are numbered so that cross referencing within and between sections, subsections, and procedures can be done easily and specifically.

If one part of a procedure is referenced (i.e. see 3.2.2.2), the first number indicates the section and the second number indicates a specific grouping. The third number indicates a particular method while the fourth number refers to a certain part of that method.

An example of the numbering system is as follows:

Section 3.2.2.2

Sample Processing and Laboratory Analysis
Chemical Methods
pH
Comments

1.2 UNITS AND CONVERSIONS

To Convert	To	Multiply By
acre	hectare	0.4047
centimeters	inches	0.3937
feet	meters	0.3048
gallons (U.S. liq.)	liters	3.785306
grams	pounds	0.002205
grams	kilograms	0.001
hectare	acre	2.471
inches	centimeters	2.540
kilograms	grams	1000.0
kilometers	miles	0.6214
lb/acre	ppm	0.5
liters	gallons (U.S. liq.)	0.2641794
liters	milliliters	1000.0
miles	kilometers	1.609
milliliters	liters	0.001
millimeters	meters	0.001
meters	feet	3.281
meters	millimeters	1000.0
ounces	liters	0.02957
pounds	grams	453.6
ppm	lb/acre	2.0
section (1 sq. mile)	acres	640
°F	°C	5/9 (°F-32)
°C	°F	(9/5 °C) + 32

1.3 PREPLANNING TOTAL MINING OPERATION

1.3.1 Acid-Base Account

In the humid areas of the United States, the toxicity associated with acid results largely from the oxidation of iron disulfides. This process takes place when earth disturbance activities such as mining (Temple and Koehler, 1954; Hill, 1970) and highway construction (Miller et al., 1976) expose iron disulfides to the atmosphere. Since the public in the United States has supported legislation that acid-toxic or potentially toxic materials (a source of pollution) will not be left exposed, the need for a basis to evaluate overburden materials arose.

Acid-base accounting is a dependable criterion by which overburden materials can be evaluated. An acid-base account consists of two measurements: (1) total or pyritic sulfur and (2) neutralization potential. The accounting balances maximum potential acidity (from immediately titratable sources plus sulfuric acid equivalent calculated from total sulfur) against total neutralizers (from alkaline carbonates, exchangeable bases, weatherable silicates or other rock sources capable of neutralizing strong acids as measured by the neutralization potentials).

The total or pyritic sulfur content (see 3.2.4) accurately quantifies potential acidity of materials when all sulfur is present as a pyritic mineral. When gypsum is found in an overburden sample or the materials are weathered, sulfur occurs in the form of sulfates. Samples high in organic carbon usually contain organic sulfur. When part of the sulfur occurs in nonacid-producing forms, the maximum potential acidity as calculated will be too high. It is for this reason that such calculations are referred to as maximums and that in doubtful cases appropriate acid and water leachings should be made to rule out those forms of sulfur which do not produce acid (see 3.2.6). Then from the stoichiometric equation of pyrite oxidation, the maximum potential acidity can be calculated in terms of calcium carbonate equivalent. Overburden material containing 0.1% sulfur (all as pyrite) yields an amount of sulfuric acid that requires 3.125 tons of calcium carbonate to neutralize one thousand tons of the material. The neutralization potential (see 3.2.3) of overburden materials, the second component of a net acid-base account, measures the amount of neutralizers present in the overburden materials. This measurement is found by treating a sample with a known amount of standardized hydrochloric acid, heating to assure complete reaction, and titrating with a standardized base. The result is then expressed in calcium carbonate equivalents. When balanced against acidity from the total sulfur measurement, a net acid-base account can be made.

From the acid-base account, potentially toxic material is defined as any rock or earth material having a net potential deficiency of 5.0 tons of calcium carbonate equivalent or more per 1000 tons of material. The 1000 tons is based on the assumption that an acre plow-layer contains 2 million pounds of soil. Regardless of the acid-base account, materials which have a pH of less than 4.0 in a pulverized rock slurry in distilled water are defined as being acid-toxic.

The choice of the deficiency of 5 tons of calcium carbonate equivalent per 1000 tons of material as the division between toxic and non-toxic material obviously is arbitrary. However, when applied to the large number of samples studied during the past several years of minesoil research at West Virginia University, it corresponds to other supporting laboratory information about these samples as well as to extensive field experiences with minesoils developing in the different rock types. If rock or soil samples were defined to be toxic at much lower calcium carbonate equivalent deficiencies than 5 tons per 1000 tons, we would be declaring many of our native soils to be toxic. On the other hand, with deficiencies much greater than 5 tons per 1000 tons, toxic concentrations of plant-available aluminum and pH values below 4.0 often develop rapidly.

Rock type is incorporated with the acid-base account because it is useful to categorize the materials which comprise coal overburdens. Knowledge of the rock types can provide an estimate of the texture and base status of a future minesoil, as well as stability of rock fragments. For example, sandstones containing moderate amounts of pyrite and lacking sufficient neutralizers become active acid producers when exposed to the atmosphere.

The properties previously discussed are represented graphically in Figure 1. There are two zones of acid-toxic materials (the 16.2 to 17.1 m and the 20.7 to 21.6 m depths) indicated by pH values of less than 4.0. Both zones contain enough sulfur to continue to overwhelm the small amount of neutralizers present. Thus, these materials have the potential for remaining acid-toxic unless large amounts of neutralizers (50 and 80 tons calcium carbonate equivalent per 1000 tons of material, respectively) are added. In addition, there is a zone of potentially toxic material at a depth of 13.4 to 16.2 m and two zones below the 23 m depth (underlying the first coal and overlying the bottom coal), which are defined by a calcium carbonate deficiency of more than 5 tons per 1000 tons of material even though the pH is above 4.0.

Non-toxic zones, which exhibit varying amounts of excess neutralizers, exist from the surface to a depth of 13.4 m, from the 17.1 to 21 m depth, and from the 24.4 to 25.4 m depth. These materials can be removed and replaced in sequential order, selectively blended before replacement, or totally blended before replacement. Other methods of handling the overburden materials would include utilization of the limestone, after crushing, as a source of neutralizers to be blended with the potentially toxic materials.

The acid-base accounting method provides a useful tool for evaluating overburdens in the humid areas of the United States, since it is useless to look for plant toxicities from elements such as aluminum, boron, etc., until the acid problem is eliminated.

1.3.2 Geologic and Pedologic Considerations

The decision of management to open and operate a strip mine is based on numerous considerations involving mining engineers, geologists, agronomists and other reclamation specialists. Initial considerations of prospective stripping areas include accessibility, proximity to markets, uniformity,

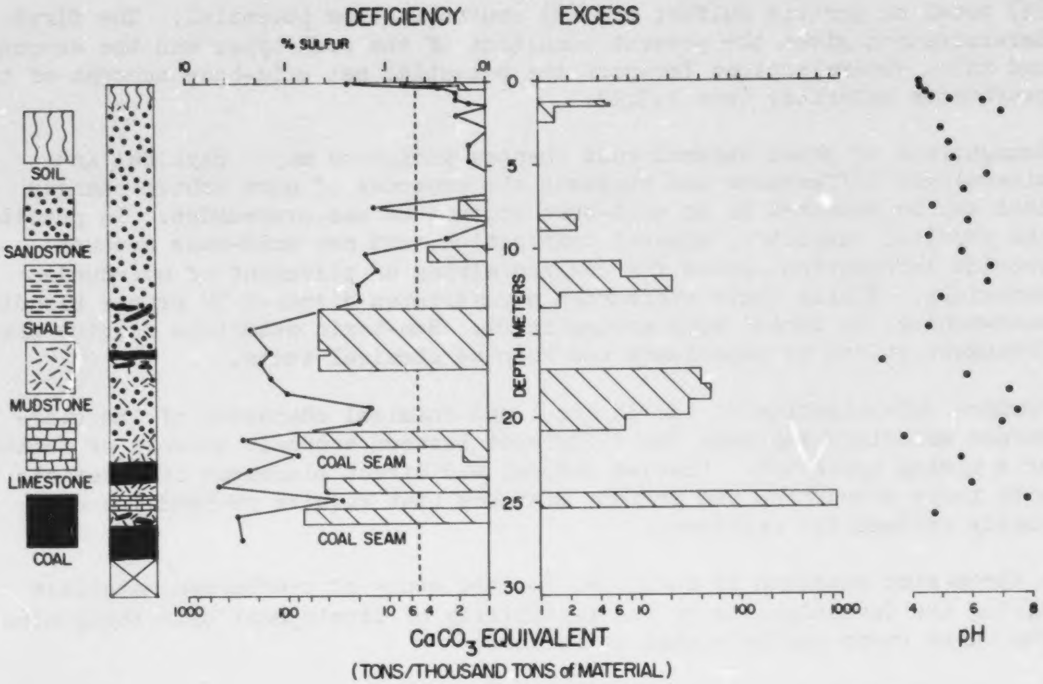

Figure 1. Acid-base account of the Kentucky no. 11 and no. 12 coal overburden.

thickness, quality and quantity of coal seams, and physical and chemical characteristics of the overburden materials for use in reclamation. In recent years the prospect of successful reclamation of mined lands resulting from selective placement of non-toxic, nutrient-rich overburden materials has become increasingly important in the decision to locate and develop new strip mines.

Coal-bearing overburden bedrock in the Appalachian and Midwestern coal basins commonly consists of a complex series of mudrocks and sandstones interbedded with generally thinner, more regular beds of limestone, carbolith, coal, and coal undersoil. The predominate mud and sand rock stratigraphic units often change rapidly in short distances (laterally and/or vertically) as compared with the chemical (limestone) and organic (coal) deposits. Usually, the rock units are mixtures in varying proportions of the common sedimentary rock types. In addition, trace amounts of heavy minerals occur in all sedimentary rocks. Of these minerals, chemical studies of overburden materials are most concerned with concentrations of the potentially acid-forming heavy metallic minerals, mostly iron disulfide, called pyritic minerals.

The immediate and maximum acid-making potential of the rock types in the overburden, is assayed by determining: (1) pH of the pulverized rock paste;

(2) total or pyritic sulfur; and (3) neutralization potential. The first determination gives the present condition of the rock types and the second and third determinations forecast the potential net acid-base account of the overburden materials (see 1.3.1).

Recognition of gross lateral rock changes indicates major physical and mineralogic differences and suggests the presence of more subtle changes that may be measured in an acid-base account of the overburden. In practice, the physical character, mineral composition, and net acid-base account provide information needed for desired mixing or placement of overburden materials. Highly toxic overburden necessitates disposal by proper blending, sandwiching, or burial with neutralizers. Non-toxic materials require proper treatment guided by experience and adapted chemical tests.

Advance determination of the physical and chemical character of the overburden materials may mean the difference between economic success or failure of a mining operation. Planned removal and direct placement of materials with known properties can prevent mistakes that require re-handling or costly reclamation practices.

A three-step approach is suggested for the study of overburden materials during the investigation of the feasibility of development of a strip mine. The three steps may be stated as follows:

1. Geological and soil reconnaissance.
2. Regional study of physical and chemical properties of soil profiles and rock units of overburden materials as well as underlying coals.
3. Detailed analyses of appropriate samples to determine important physical and chemical characteristics of soil horizons and rock units of the overburden at promising sites.

Geological and soil reconnaissance in the area of interest consists of a review of information from private, state and federal sources. Available information is generally confined to modern standard soil surveys, 7.5 minute topographic maps, county geologic reports, physical and mineralogical descriptions of rock units, chemical analyses of coals, and location of underground mines, surface mines and coal prospects. Collection and analysis of selected dominant soil profiles and rock units of prospective overburden materials in the area of interest may offer early clues to future land use and reclamation opportunities.

Delineation of the physical and chemical relationships of overburden materials and associated coals is depicted by construction of generalized cross sections similar to those in northern West Virginia and western Maryland as illustrated in EPA reports 670/2-74-070 (Smith et al., 1974) and 600/2-76-184 (Smith et al., 1976). Sampling of correlative rock units can usually be obtained at exposures, abandoned and active surface mines, or from drill cuttings and cores along or adjacent to the line of cross section in areas of contour stripping. Cores and highwalls of active strip mines are usually the only source of samples in environs of area stripping, which are often covered with unconsolidated deposits (e.g. loess, glacial till, and outwash).

Collection of more closely spaced detailed geological and chemical data continues during exploration of the proposed stripping site and also during the stripping operation. Sources of geological information and samples are cores, churn drill cuttings, blast hole cuttings, and exposures of excavations and highwalls. Sampling sites should be spaced to give a three-dimensional coverage of the area of stripping. The geologist-pedologist describes the soil and lithologic units exposed or penetrated by the drill in some detail. He samples the rock section either in arbitrary 30 cm intervals or other appropriate intervals (see section 2.2) for chemical and physical analysis. Three-dimensional illustrations such as ribbon diagrams (showing soil profiles and lithologic units plus acid-base or other relationships) or isopachous maps (contouring thickness of separate lithologic units and weighted averages of acid-base relationships) can provide interesting artistic views of problem areas to be encountered prior to stripping. A similar exercise is the construction of a series of intersecting cross sections illustrating the overburden information derived from all available sources. Such cross sections show a combined geologic and chemical accounting of the overburden materials throughout the area to be stripped. Intersecting cross sections provide the operator with a pictorial view of the physical and chemical characteristic of the overburden rock in advance of strip mining and guides the operator in the handling and segregating of materials to ensure favorable minesoils and economical reclamation for intended land use.

Following mining, the young minesoils are appropriate for classification into classes based on properties. The mapping of soil classes and phases is simplified by good advance information about overburdens and their placement during mining. Short-term treatment and long-range management follow established patterns once minesoil mapping units have been established and delineated.

SECTION 2

FIELD CLUES AND SAMPLING METHODS

2.1 WHAT TO LOOK FOR AND MEASURE IN THE FIELD

2.1.1 Summary

Procedures in this section aid the field observer. As one looks at an overburden column, prominent characteristics observed include changes in color, soil horizons, rock types and special features such as nodules, layers, faults and coatings. As seen in Figure 2, the sandstone unit at the surface is divided due to the degree of physical and chemical weathering. The high chroma (brown) shows where oxidation of iron has occurred. This upper zone will be lower in sulfur due to oxidation and leaching. It is generally more porous and less consolidated than the underlying low chroma (gray) unoxidized rock. Most of the carbonates that were present in the oxidized rock have been removed by leaching. Directly underlying the sandstone unit is a low chroma mudrock or shale, which may or may not contain carbonates. A layer of black carbolithic mudrock directly overlies the coal. The high carbon material, indicated by a color value of 3 or less, may be high in acid-producing sulfur.

The Pittsburgh-Redstone coal overburden, as seen in Figures 3 and 4, is high in calcareous materials. A bed of limestone, the light gray layer, occurs between the coal seams. This limestone is stained with yellowboy (iron oxides) where sulfate-rich waters have been neutralized and precipitated out. The yellowboy color varies depending on the form and amount of iron present. The yellowboy staining can be seen on the limestone between the coal seams in Figure 3 and also where the sulfate-rich waters are coming from the base of the Pittsburgh coal and draining over a calcareous mudstone in Figure 4. A rock type comparison can be made between the overburden in Figure 2 which is comprised mainly of sandstone with some mudrock or shale and the overburdens in Figures 3 and 4 which are predominantly limestones and calcareous mudstones, mudrocks, and shales with some sandstone.

The classification of soil horizons and rock type and the determination of color are given in the following procedures. To aid in the determiniation of soil horizons and rock types and to predict their useful properties, methods for rock hardness, detection of calcareous materials, and estimation of rock and soil textures are included. Special interest features (such as presence of pyrite, mica, gypsum, epsomite, Fe-Al sulfates and others) assist the observer in predicting potentially favorable or unfavorable materials in the overburden for selective placement and use during reclamation. At the

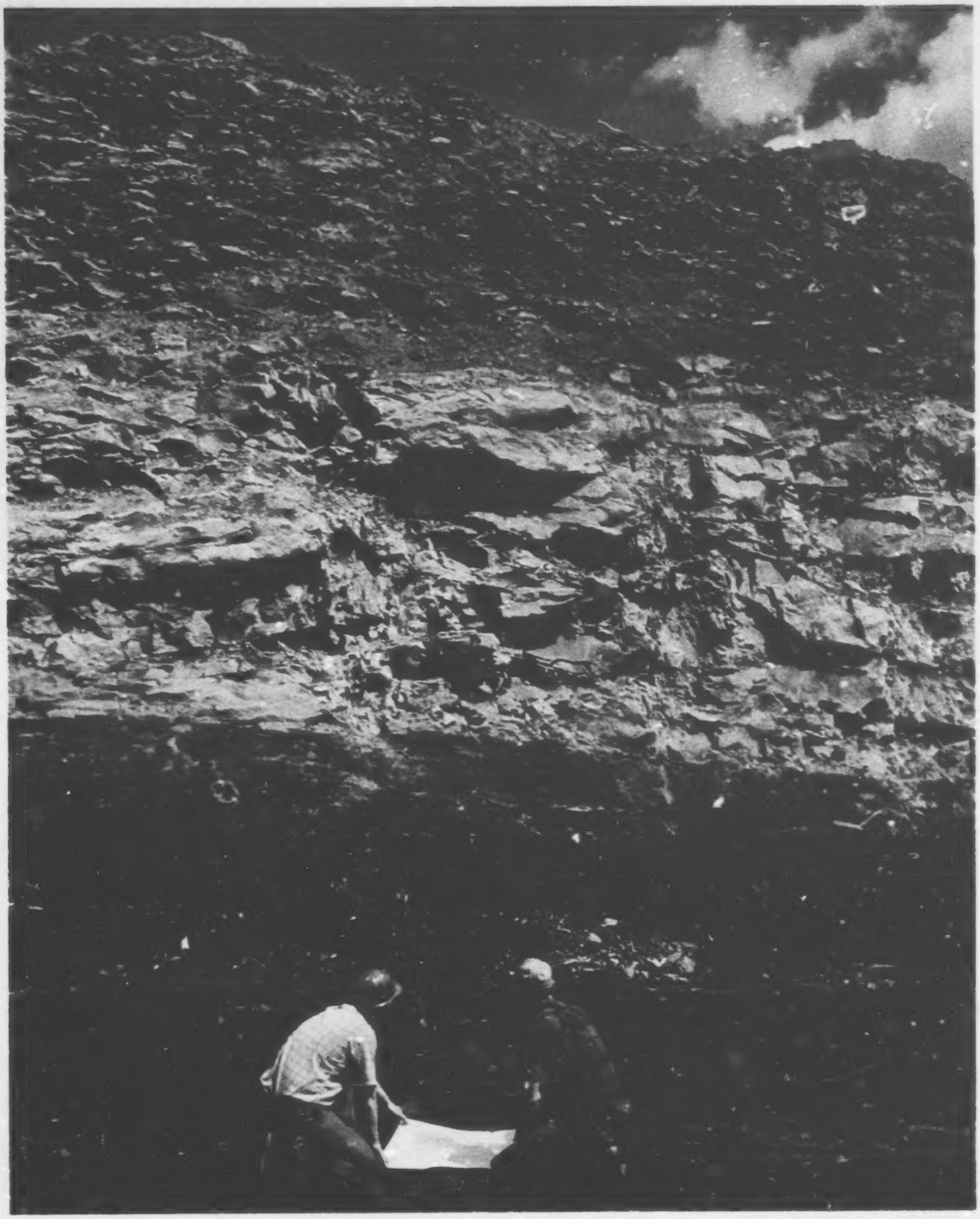

Figure 2. Highwall showing high and low chroma color characteristics.

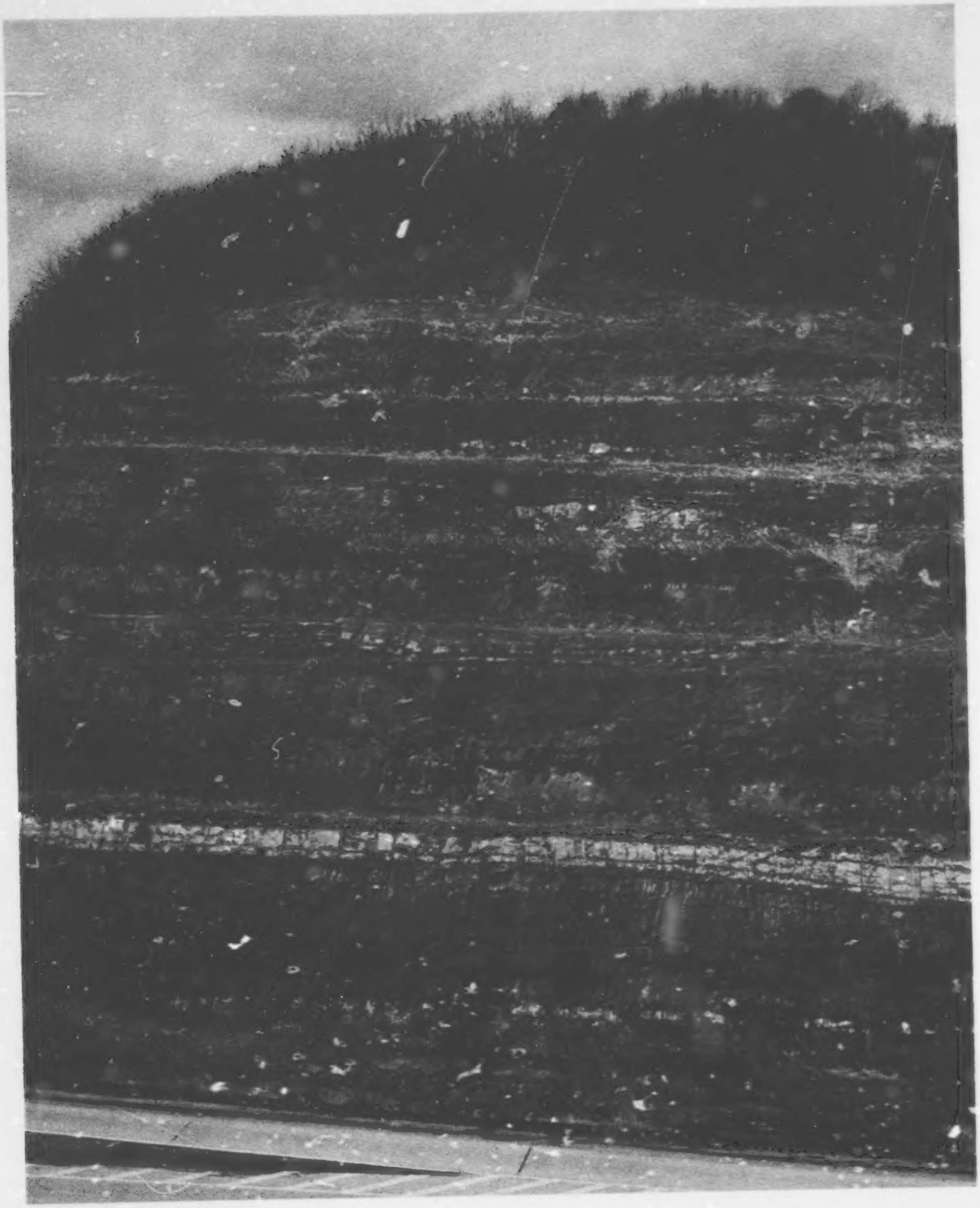

Figure 3. Pittsburgh-Redstone overburden showing yellowboy staining on the limestone between the coal seams.

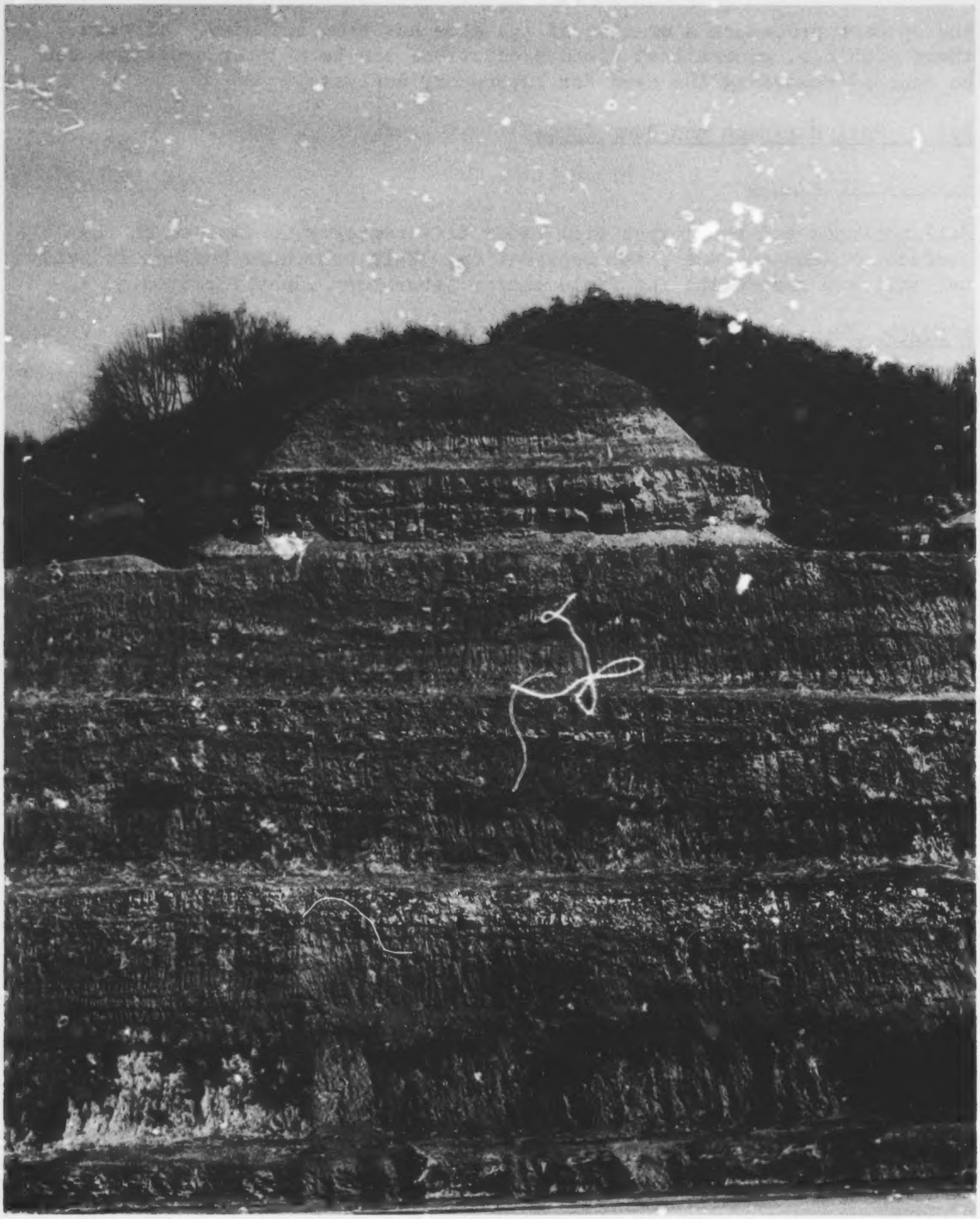

Figure 4. Pittsburgh-Redstone overburden showing extreme yellowboy staining on a calcareous mudstone below the Pittsburgh coal seam.

end of each procedure a meaning of the clue has been included. Through these meanings, generalized field predictions can be made or decisions can be reached regarding the need for laboratory analyses.

2.1.2 Soil Horizons and Rock Types

2.1.2.1 Principle--

Soil horizons and rock types will react differently when exposed on the surface or near-surface after reclamation. This procedure defines individual soil and rock units for field and/or laboratory identification.

2.1.2.2 Comments--

Both soils and rocks must be examined on a freshly exposed surface. The following characterizations of each soil and rock type will aid in their identification:

1. Soil Horizon 1 is the surface layer which is usually darkened by organic matter. It is the zone of maximum biological activity (i.e., it will have the most plant roots; the most earthworm activity) and the zone of maximum accumulation of organic matter.

2. Soil Horizon 2 lies between Horizons 1 and 3 and often is referred to as the "subsoil". It will have some plant roots and earthworm activity, but less than the overlying Horizon 1. Horizon 2 may contain a zone of clay accumulation, which should be favorable in a coarse textured soil and unfavorable in a fine textured soil.

3. Horizon 3 is a zone of weathered rock or earthy material. It is unconsolidated material with little or no biological activity. This horizon will often have individual rock fragments larger than 2 mm. Horizon 3 extends down to consolidated, intact bedrock or a depth of 1.5 m (5 ft) whichever is shallower. Horizon 3 may contain a fragipan.

4. A fragipan is a dense, firm layer of intermediate texture that impedes free movement of air and water down through the soil and restricts root growth. Plant roots cannot branch out in this layer and often grow laterally along its top. When crushed between thumb and finger, a dry piece of this layer shatters abruptly rather than crumbling gradually. The fragipan becomes extremely hard during the dry season and may be difficult or impossible to penetrate with a soil tube or to crush with thumb and forefinger.

5. Earthy material (EM) is a broad term for any unconsolidated material between a depth of 1.5 m (5 ft) and consolidated bedrock. It may be similar to horizon 3 in composition and appearance.

6. Drift is a broad term for glacial deposits.

7. Till is unstratified and unsorted drift deposited directly by glacial ice. Till consists of clay, silt, sand, gravel, and boulder-size particles of varied rock types which can be intermixed in any proportion.

8. Outwash (OW) was deposited by melt-water streams beyond active glacial ice. In contrast to till, outwash is stratified and sorted.

9. Loess is a homogeneous, unindurated deposit consisting predominantly of silt-size particles, with smaller amounts of very fine sand and/or clay-size particles. Loess may or may not be stratified.

10. Sandstone (SS) contains more than 50 percent sand-size (less than 2 mm and greater than 0.05 mm in diameter) particles. The particles are predominantly quartz and may be cemented with silica, iron oxide, carbonates, or clays. Qualitative modifiers such as calcareous, argillaceous, micaceous, and pyritic, for example, are used when they seem to add useful information.

11. Mudrock (MR) is a broad term for a sedimentary rock dominated by silt-size and/or clay-size particles. The term is used when a rock cannot be definitely distinguised as either a mudstone or shale. Mudrock can be further subdivided into hard mudrock (moist hardness greater than 2.5) or normal mudrock (moist hardness less than 2.5). Mudrock may contain as much as 50 percent sand-size particles if properties are judged to be dominated by silt and/or clay. Mudrocks may contain any proportion of carbonates so long as properties are dominantly silt and/or clay when rubbed in water.

12. Mudstone (MS) is a non-fissile mudrock dominated by silt-size and/or clay-size particles. Mudstones have a moist hardness of less than 2.5. They differ from shale because of their non-fissile nature. Mudstones may contain as much as 50 percent sand-size particles if properties are judged to be dominated by silt and/or clay.

13. Shale (SH) is a mudrock that appears predominantly fissile (having a tendency to split along parallel planes into thin layers). These layers must be less than 5 mm thick. Shales can be further subdivided into hard shale (moist hardness greater than 2.5) and normal shales (moist hardness less than 2.5). They differ from mudstones because of their fissile nature.

14. Limestone (LS) is a sedimentary rock consisting dominantly of calcium carbonate. On a freshly exposed surface, limestone will react with a noticeable "fizz" upon application of dilute hydrochloric acid. Limestones must have a moist hardness of greater than 2.5, thus distinguishing them from calcareous mudstones. When powdered, the powder will have a Munsell color value of 7 or greater. Some limestones are dolomitic due to substitution of magnesium for some of the calcium. Dolomitic limestones (or dolomite) will only react with cold dilute hydrochloric acid when applied to the rock powder.

15. Chert, Flint, and Jasper are rocks consisting dominantly of amorphous silica or extremely small (cryptocrystalline) quartz and hard (6.5 to 7.0 on Moh's scale) enough to scratch glass or an ordinary knife blade.

16. Carbolith (Carb) is a name that has been coined (Smith et al., 1974) to cover dark colored sedimentary rocks that will make a black or very dark (Munsell color value of 3 or less) streak or powder. Rocks under this name include coal not scheduled for mining, impure waste coal, bone coal, high-carbon shales, and high-carbon muds. In general, such rocks will contain at least 25 percent carbonaceous matter oxidizable at 350-400°C.

17. Intercalate (I) is a term coined for use in this manual to describe rocks which contain at least two different rock types that are so intimately interlayered or "intercalated" that they cannot conveniently be sampled separately. Intercalates have at least three or more layers within a 13 cm (5 in) measured section. This rock type can be defined in greater detail by listing in order of abundance some or all of the kinds of rocks included. Commonly only two or three types of rock will be listed to suggest the dominant properties of an Intercalate (e.g. I-SS/MS, I-SS/MR/Carb).

2.1.2.3 Chemicals--

Hydrocholoric acid (HCl), 1 part acid to 3 parts water: Dilute 250 ml of concentrated HCl to a volume of 1 liter with distilled water.

2.1.2.4 Materials--

1. Shovel.

2. Rock hammer.

3. Soil knives (any kind of knives, nails, knitting needles, pencils, or pointed objects can be substituted).

4. Dropper bottle (for holding the acid).

5. Wash bottle.

6. Munsell color book.

7. Record book.

8. Ruler or tape measure.

9. Hand lens, 10 power.

2.1.2.5 Procedure--

2.1.2.5.1 For Soils--These steps are used for the determination of soil horizons.

1. Examine freshly exposed soil profile. NOTE: If a freshly exposed profile is not available, a pit can be dug or a core taken of the profile. If a profile does exist, it can be cleaned off with a shovel to expose a fresh surface.

2. Examine profile for the point of separation of horizons 1 and 2 (see 2.1.2.2) and mark point with a soil knife.

3. Examine profile for the point of separation of horizons 2 and 3 (see 2.1.2.2) and mark point with a soil knife.

4. Beginning at the original land surface, record depth of each horizon.

5. Record depth to bedrock or 150 cm (5 ft) whichever comes first. NOTE: If depth to bedrock exceeds 150 cm (5 ft), record thickness of earthy material (see 2.1.2.2).

6. Examine profile for presence of a zone of clay accumulation (see 2.1.2.2). Record depth from surface and thickness if found to exist.

7. Examine profile for presence of a fragipan (see 2.1.2.2). Record depth from surface and thickness if found to exist.

8. Record color of each horizon (see 2.1.3).

9. Record texture of each horizon (see 2.1.8).

10. Record presence of any nodules, concretions, or any other features deemed necessary to detail the profile.

2.1.2.5.2 For Rocks--These steps are used for the determination of rock type.

1. Examine a fresh surface of the rock. NOTE: This can be accomplished by breaking the rock with a rock hammer.

2. Test rock for hardness (see 2.1.4).

3. Test for presence of carbonates with 1:3 HCl (see 2.1.5).

4. Using a knife, scrape the rock to form a powder. Determine powder color (see 2.1.3). NOTE: The powder color can be taken of some rocks by streaking the rock on a streak plate (unglazed porcelain plate) and determining the color of the streak.

5. Using data obtained in steps 2-4, determine and record rock type (see 2.1.2.2).

6. Record presence of pyrite and/or mica (see 2.1.6) as well as any other rock features (see 2.1.7).

2.1.2.6 Meaning of Clue--

1. If horizon 1 is 25 cm (10 in) or more in thickness having a moist color value and chroma of 3.5 or less, it will be high in soil organic matter, can be high in plant nutrients, and generally have favorable properties with respect to tillage and water relationships.

2. Fragipans make unfavorable soil material.

3. Zone of clay accumulation could be unfavorable soil material, especially where clay content exceeds 35 percent.

4. Drift and till can contain members of any size fraction from boulders to clay. Individual characteristics will determine their use.

5. Outwash, if mixed with suitable "fines," may have good soil potentials.

6. Loess will have a favorable soil texture and usually is calcareous. Soils developed in loess that have been leached can be neutral to strongly acid.

7. Sandstone can have a textural range from very coarse to loamy and can be pervious. Proportions and porosity of coarse fragments are important variables that depend on strength of cementation and mineralogy.

8. Mudrock can have the properties of a mudstone and/or shale. Soils formed from mudrock will be of a medium to fine texture and, depending on hardness, may or may not produce coarse fragments. Calcareous mudrock should be considered for its neutralizing potential.

9. Mudstones will form soils having a medium to fine texture. In some cases, high-alumina clays are abundant, and resulting soils have relatively high anion-exchange but low cation-exchange capacity, even though clay percentages are high. Minesoil management difficulties may occur with either silty or clayey textures because of weak structures. Plant nutrient reserves may be adequate, and carbonates may be present at any level below that of a recognized limestone or dolomite.

10. Shales can form soils having a medium to fine texture with coarse fragments in the form of chips derived from their fissile nature. Any level of carbonates and plant nutrients may be present.

11. Limestones and dolomites will persist as coarse fragments, unless broken down during mining operations. As long as limestone or dolomite remain in coarse fragments, neutralization effects will be minimal.

12. Chert, flint, and jasper, if highly weathered, may contain considerable useful porosity.

13. Carboliths are a common source of pyritic sulfur. These rocks may contain carbonates or simple or complex sulfate salts. Carboliths may be high in phosphorus which can be used as a plant nutrient if the toxic acids can be neutralized. Since carboliths have a color value of 3 or less, they will absorb heat which can be detrimental in hot weather and favorable in cold weather until well vegetated.

14. Intercalates, by definition, are combinations of any of the above rock types and would have the characteristics of the incorporated rock types.

2.1.3 Color

2.1.3.1 Principle--

A standard color system is required for uniformity of description among field workers. The Munsell Soil Color Charts are standards which subdivide color into three measureable elements: hue, value and chroma.

In these color charts, hue is the dominant spectral color (red, yellow, green, blue, and purple) and is related to wavelength of light; value is the measure of lightness or darkness and is related to total reflected light; and chroma indicates purity or strength of color (or departure from neutral of the same lightness).

2.1.3.2 Comments--

The quality and intensity of light affects the visual impression of color from the standard color chips and the sample. When using the color standards in the field or laboratory, it is important that the quality of light be similar to the white light of mid-day and the amount of light be adequate to visually distinguish between the color chips. Color measurements made in the field during early morning or late evening and during a hazy overcast day will not be precise.

Color values are usually lower when samples are moistened as compared to air-dry. Color measurements are made on air-dry, powdered (less than 60 mesh) samples in the laboratory and on a freshly exposed surface in the field.

2.1.3.3 Chemicals--

None required.

2.1.3.4 Materials--

1. Munsell Soil Color Charts (available from Munsell Color Division, Killmorgan Corporation, Baltimore, Maryland 21218).

2. Spatula.

2.1.3.5 Procedure--

NOTE: If powdered (less than 60 mesh) sample is used instead of soil ped or rock fragments, place 0.5 g of sample on the tip of a spatula and omit steps 1 and 2.

1. Break soil ped or rock fragment in half.

2. Use a freshly exposed surface to determine color. NOTE: In the case of more than one color being present, select the dominant color for color determination. Record the secondary color(s) as "mottles."

3. Compare the sample to the 10YR chips. NOTE: If the sample is judged to be more red, compare sample to chips with a more red hue. If the sample is judged to be more yellow, compare sample to chips with a more yellow hue. If the sample is judged to have a chroma less than 1, compare sample to neutral chips.

4. After selecting the proper hue, match the sample to the chip to which it most closely corresponds.

5. Record the hue, value and chroma. NOTE: The color hue is the number found in the top right corner of the color page, the value is the number to the left of the row in which the color chip was selected, and the chroma is the number at the bottom of the color chip column. Color is recorded as hue then value then chroma (e.g. 10YR 6/4).

2.1.3.6 Meaning of Clue--

In rock material, hue can be used in a very general way as a clue to indicate rock quality. A striking example of a favorable minesoil material having a readily distinguished color hue and chroma is the dusky red shales and mudstones common in western and northwestern West Virginia.

Value can be used to readily distinguish highly carbonaceous black shales from true gray shales that appear black to the casual observer. Bonecoal, roof shales, and other dark or black appearing rocks frequently contain significant amounts of pyrite and may be a source of extreme sulfuric acid acidity unless neutralizers are present. The field clue to such material is a black (value of 3 or less on any Munsell hue) streak when rubbed on an unglazed porcelain plate or hard white rock such as chert or when the rock is powdered by scraping with a knife. Dark colored clay or silty shales that are low in carbon, on the other hand, are medium or light gray (Munsell color value of 4 or higher) when powdered.

Chroma is one of the most easily recognized color attributes, and can be used to recognize many soil and rock features. It is now well established that minesoil developing in overburden from the intensely weathered zone below the original land surface is safe from pyritic sulfur (pyrite, marcasite, and chalcopyrite) and extreme acidity. This zone commonly is 6 m (20 ft) deep or deeper in West Virginia, depending on lithology, degree of structural fracturing of the rock, and position of the water table. Brown and yellow rock colors (chroma 3 or higher on Munsell Soil Color Charts) as typified by materials from the weathered zone, provide useful clues to safe materials regardless of their position in the stratigraphic section. However, absence of high chromas in near-surface soils and rocks can result from intense leaching of iron oxides or (in soils) from impeded drainage which causes iron reduction. The low chroma imparted to the surface of highly leached materials in soils and near-surface rocks can be distinguished readily from low-chroma rocks below depth of iron oxidation. Low chromas (gray colors) caused by leaching or impeded soil drainage occur on rock or soil ped exteriors. In contrast, low chromas occur on the interiors of unoxidized sandstones or shales. Color chroma has proven reliable as a field clue particularly with many sandstones. Freshly broken rock surfaces with chromas of 3 or higher (hand specimen or pulverized sample) indicate negligible percentages of pyritic sulfur. Chromas of 2 or less often correspond with sufficient pyrite to cause pH below 4.0 and biotoxic reactions unless neutralizers are abundant.

Darker color values of undisturbed surface soils commonly indicate high organic matter content. A moist soil value and chroma of less than 3.5 indicates a mollic, umbric, or anthropic surface horizon.

2.1.4 Determination of Rock Hardness

2.1.4.1 Principle--

Hardness is the resistance of a mineral or rock to scratching. The numerical value for hardness is based on Moh's hardness scale. Moh derived a scale where the softest mineral, talc, is number 1 and the hardest mineral, diamond, is number 10. All minerals (and rocks) fall within this range of 1 to 10 depending on hardness.

2.1.4.2 Comments--

Three ranges of hardness (less than 2.5; 2.5 to 5; greater than 5) based on Moh's scale are used in this procedure. These ranges are determined in the field by using the hardness of the fingernail as 2.5 and the steel of a pocket knife as a little over 5.

Care must be taken to insure that a powder, and not the breaking off of individual grains, is being formed when a hardness "standard" (fingernail or steel knife) is scratched against the rock fragment. This is especially true with sandstones. NOTE: Care must be taken to insure that the "standard" is not scraping off on to the rock. A visible groove should be evident in the rock surface if it is scratched.

2.1.4.3 Chemicals--

None required.

2.1.4.4 Materials--

Steel knife.

2.1.4.5 Procedure--

1. With the steel knife try to scratch the rock fragment. If no scratch occurs, record hardness as greater than 5. If a scratch occurs, continue to step 2.

2. With a fingernail try to scratch the rock fragment. If no scratch occurs, record hardness as 2.5 to 5. If a scratch occurs, record hardness as less than 2.5.

2.1.4.6 Meaning of Clue--

As a general rule: the harder a rock; the better it will withstand weathering and form coarse fragments.

2.1.5 Presence of Calcareous Material

2.1.5.1 Principle--

Calcium carbonate is the major constituent in limestone; however, soils and other rock types can also contain calcium carbonate. The addition of cold dilute hydrochloric acid to a sample containing calcium carbonate causes the following reactions to occur:

Calcium carbonate + hydrochloric acid forms calcium chloride + carbonic acid

$$CaCO_3 + 2HCl = CaCl_2 + H_2CO_3$$

Carbonic acid will further disassociate to water + carbon dioxide

$$H_2CO_3 = H_2O + CO_2$$

Since carbon dioxide gas is released, a noticeable effervescence (bubbling) and even an audible "fizz" occurs indicating the presence of carbonates.

2.1.5.2 Comments--

The particle size of the material is reduced by scraping the rock fragment with a knife or other tool to form a powder allowing more surface area to become available for reaction with the acid. Calcareous material, which may not have been detected previously, may now be detected.

Care must be taken to insure that the acid is reacting with the rock or soil and not with a calcium carbonate coating.

2.1.5.3 Chemicals--

Hydrochloric acid (HCl), 1 part acid to 3 parts water: Dilute 250 ml of concentrated HCl to a volume of 1 liter with distilled water.

2.1.5.4 Materials--

1. Dropper bottle.

2. Knife.

2.1.5.5 Procedure--

1. Add one or two drops of cold hydrochloric acid to a fresh surface of the sample. NOTE: Presence of calcium carbonate ($CaCO_3$) is indicated by a bubbling reaction or audible "fizz."

2. If no reaction occurs, scrape the surface with a knife or other tool to produce a powder.

3. Add a drop of cold hydrochloric acid to the powder and check for presence of carbonates as stated in step 1.

2.1.5.6 Meaning of Clue--

Our results indicate that at least 20 tons $CaCO_3$ equivalent per 1000 tons of material is present if a noticeable reaction occurs.

2.1.6 Determining Rock Texture and Presence of Pyritic and Micaceous Material Using Ten Power Hand Lens

2.1.6.1 Principle--

Pyrite and mica are common minerals in sedimentary rocks. Using a ten power hand lens for magnification, the presence of small pyrite grains, mica flakes, or inclusions may be detected or confirmed. Individual mineral grains may be seen for texture observations.

2.1.6.2 Comments--

Pyrite is commonly found in crystal clusters with many faces. It has a metallic look and is usually pale brass-yellow. However, it may appear to be black due to weathering or to extremely small particle size.

Mica may range in color from pale golden brown to black. It usually appears as flakes in a rock, sometimes along bedding planes. Pyrite can be distinguished from mica, since pyrite is opaque and will glisten on all surfaces of the crystal whereas mica will only glisten on one surface as the sample is tilted.

Individual mineral grains in the size range of coarse silt or coarser can be detected.

2.1.6.3 Chemicals--

None required.

2.1.6.4 Materials--

1. Hand lens, ten power.

2. Hammer.

2.1.6.5 Procedure--

1. View surface of sample with ten power hand lens.

2. Examine for presence of pyrite or mica (see 2.1.6.2).

3. Examine texture of sample to detect individual mineral grains in the size range of coarse silt or coarser.

4. Break the rock with a hammer to expose a fresh surface. Repeat steps 2 and 3.

2.1.6.6 Meaning of Clue--

Since pyrite may weather to sulfuric acid, rocks containing pyrite may indicate zones of potentially toxic materials in the overburdens. Laboratory analyses would be needed to verify this implication.

Micas affect the weathering potential of a rock. Rocks high in mica (especially if the mica is in layers within the rock) usually tend to weather rapidly.

Texture is important in sampling and identifying rock types, especially in classifying sandstones by texture.

2.1.7 Other Soil and Rock Features

2.1.7.1 Principle--

More detail can be added to the overburden material description by noting the taste, smell, and presence of lenses, minerals, fossils, concretions and nodules. Taste and smell can indicate the presence of Fe-Al sulfates and epsomite or gypsum. Lenses, which can be between rock types or within a rock type, show a change in mineralogy. By looking at concretions, nodules, and plant and animal fossils, zones of carbon or calcareous material may be detected.

If any of these features exist in an overburden, their presence should be recorded.

2.1.7.2 Comments--

Laboratory procedures may be required to determine the presence of gypsum, epsomite, the Fe-Al sulfates, as well as the mineralogy of the concretions and nodules. Lenses, which are not detected in the field, may be found to exist after drawing the stratigraphic cross-sections (see 1.3.2) of the area.

Definitions of terms used in this procedure can be found in the glossary.

2.1.7.3 Chemicals--

None required.

2.1.7.4 Materials--

None required.

2.1.7.5 Procedure--

1. Taste and smell - The presence of simple or complex Fe-Al sulfates (usually white, gray or reddish in color) can be detected by its metallic taste and smell. The smell is similar to that of a brass door knob. Epsomite (colorless to white, but may vary due to impurities) can be detected by its bitter, non-metallic taste.

2. Lenses - Lenses may occur between or within soil and rock types. Both lateral and vertical extent should be determined and recorded.

3. Minerals - Record the presence of minerals or crystals of minerals and their composition, especially gypsum (which is colorless to white, but may vary due to impurities, and can be scratched with a fingernail) and pyrite (see 2.1.6).

4. The presence of both plant and animal fossils and their mineral composition should be recorded.

5. Concretions and nodules - Concretions and nodules may occur within both soils and rock types. Determine and record their mineralogy, frequency of occurrence, and size.

2.1.7.6 Meaning of Clue--

1. Fe-Al sulfates, epsomite, and gypsum - Fe-Al sulfates are extremely acid. Epsomite and gypsum are neutral. Epsomite provides magnesium for plant growth. Neutralization potential (see 3.2.3) and non-HCl extractable sulfur (see 3.2.4) data should be used to determine acid producing material.

2. Lenses - Lenses show a change in soil or rock characteristics which could change the net acid-base account of a mine site.

3. Fossils - A potential carbonate and/or pyrite source may be detected in animal fossils. A potential carbon and/or pyrite source may be detected in plant fossils.

4. Concretions and nodules - A change in the net acid-base account may occur depending on the mineralogy of the concretions and nodules.

2.1.8 Soil and Minesoil Texture by Feel

2.1.8.1 Principle--

Soil texture refers to the percentages of sand, silt and clay present in a sample or layer of soil. All textures can be designated as belonging to one of three families: the clayey family (includes clay loams), the loamy family (includes silt loam), and sandy family.

2.1.8.2 Comments (adapted from USDA, 1975)--

Sand particles feel gritty when rubbed between the fingers and are not sticky or plastic when wet. Silt particles feel smooth and powdery much like flour when rubbed between the fingers and are only slightly plastic or sticky when moist. Clay particles feel smooth and are sticky and plastic when moist.

A sample of the clayey family will have 35 percent or more clay content with the remaining fraction composed of silt and sand. The feel may be smooth or slightly gritty depending on the relative proportions of sand and silt present. The moist sample will feel plastic or stiff and sticky, and form a long flexible ribbon that is durable when handled, especially when the clay content exceeds 40 percent. If allowed to dry, the sample will form hard clods that are difficult to break apart with the fingers, especially with higher clay contents.

A sample of the loamy family will contain less then 35 percent clay and less than 15 percent fine sand or coarser. The moist sample may or may not be sticky and plastic or form a ribbon that breaks easily depending on silt content. The feel will be somewhat gritty if the sand fraction dominates and smooth (flourly) if silt dominates. Firm clods that can readily be crushed with the fingers will form upon drying.

In areas where soils are high in silt, the loamy family can be subdivided into a silty family and loamy family. The silty family will contain less than 15 percent sand and greater than 65 percent silt with the remaining material being clay. A moist sample will feel smooth when kneaded and will not feel gritty nor form a very good ribbon.

A sample of the sandy family will contain sands and loamy sands, exclusive of loamy very fine sand and very fine sand textures. When the moist sample is rubbed between the fingers, it will feel abrasive and no ribbon will be formed.

The adjectives skeletal or fragmental can be added to the above textural families. If particles having an equivalent diameter coarser than 2 mm make up at least 35 percent by volume of the layer being studied and contain enough fine earth to fill the larger than 1 mm interstices, the term skeletal is used. Soils dominated by stones, cobbles, gravel, and very coarse sand particles with not enough fine earth to fill the larger than 1 mm interstices are termed fragmental.

Texture by feel can be confirmed by laboratory analysis, either by the hydrometer method or the pipette method. See sections 3.4.2 and 3.4.3 for discussion of these procedures.

2.1.8.3 Chemicals--

Water (H_2O).

2.1.8.4 Materials--

Wash bottle.

2.1.8.5 Procedure--

1. Take about a teaspoon of soil in the palm of the hand.

2. Add water slowly from the wash bottle, constantly kneading the soil. Knead to the consistency of a moist workable putty. NOTE: Working the soil to the proper consistency is critical since moist soil feels different to the fingers than dry soil.

3. When the soil is at the proper consistency, rub it between the thumb and fingers. Try to press the soil into a thin ribbon.

4. Determine the soil or minesoil texture using section 2.1.8.2.

2.1.8.6 Meaning of Clue--

By taking soil and minesoil textures, the relation of particle sizes with each other can be determined. If a sample is high in clay, compaction problems can exist. Samples high in sand content can be a problem during periods of extensive drought due to low water holding capacities. Samples high in silt are more favorable.

2.2 OVERBURDEN SAMPLING AND LABELING

2.2.1 Summary

Before useful laboratory analyses can be performed, consistent sampling and labeling procedures must be utilized. Exploration cores can provide excellent samples. Since rock cores remain intact, accurate rock type depths from the surface, and thicknesses can be measured. Any vertical variation within a rock unit can be seen and noted.

If exploration cores are not available, samples at 30 cm (12 in) increments can be obtained using a blast hole drilling rig. The rock chips blown from the drill are collected on a shovel. Exact rock type, depths from the surface, and some vertical variations within rock units are lost.

Hand samples can be taken from a freshly exposed high wall if neither exploration cores nor blast hole drillings are available. Accurate depths from the surface and variations in rock units can be determined; however, the procedure of working vertical faces with ropes and ladders may be time consuming.

Materials of special interest can also be selectively sampled. Selective samples, as the term implies, are taken by hand. They are especially useful for checking variation within rocks that appear similar and for determining properties of peculiar or extreme specimens.

Once a sample is collected, it must be properly labeled to include all data about the site. Information such as site location, depth taken, rock type, and date sampled are necessary, not only to keep from confusing samples, but to locate sampled areas if the need should arise.

Since variations can exist within an overburden, the laboratory data can only reflect what exists within that particular exploration core, blast hole, hand sampled high wall column, or selective sample. The more information acquired about a site, the better an overall picture can be made of the overburden material.

2.2.2 Rock Chip Sampling From Blast Hole Operations

2.2.2.1 Principle--

Rotary drilling gives a vertical column of the overburden material. The drilling breaks the material into rock chips and compressed air brings the chips to the surface.

2.2.2.2 Comments--

Samples should be taken where overburden material is the thickest, e.g. top of a hill or farthest strip cut into a slope, to obtain the most information. The lateral distance and direction between sampled blast holes should be recorded where more than one column of rock chip samples is collected on a job. Indication of upslope or downslope from a previous sample should be recorded.

The geographical location of the sample site should be located on a U.S.G.S. 7 1/2 minute topographic map. Latitude and longitude coordinates are determined to four decimal places by using a ruler.

Blast hole drilling offers a speedy and easy method of collecting rock chip samples. Exact depth of a rock type break is lost, but relative depths can be obtained.

This procedure will not work with some center-platform types of drills. Samples from the drill bench to the surface must be taken by hand sampling (see 2.2.3.1) for a complete vertical column of the overburden material.

2.2.2.3 Chemicals--

None required.

2.2.2.4 Materials--

1. Long handled shovel (common round pointed garden shovel is adequate).

2. Container with lid, round, one pint or one quart. NOTE: One container for each foot drilled or each sample obtained is required.

3. Felt-tip pen or other marker for legibly labeling sample container.

4. Wooden crate or heavy corrugated paper carton to transport rock filled containers.

5. Drill rig, rotary bit, compressed air type (Robbins Rotary Drill Model RR-T or similar type).

6. Record book.

2.2.2.5 Procedure (revised and updated from Smith et al., 1974)--

1. Determine the number of links per foot of the drive mechanism suspension chain on the drill rig.

2. Depth increment is approximated by marking successive link pins that occur about 30 cm (12 in) apart. Use a dab of grease or other mark that will be visible through the dust. NOTE: Every sixth pin is 34 cm (13.5 in) on the commonly used Robbins drill rig. This is close enough to the suggested 30 cm depth increment, but total depths recorded should count the full 34 cm in each successive increment.

3. Begin sampling with the first 30 cm (12 in) increment drilled from the leveled bench on which the rig is parked.

4. Hold shovel under dust apron almost touching the rotary drill extension. Air-expelled rock chips are allowed to collect on the shovel as the bit lowers 30 cm (6 link pins). NOTE: If an obvious change in rock type occurs within the 30 cm (12 in) interval, the rock types should be sampled separately and depth of change recorded.

5. Place shovel-full of sample in a container. Discard any material overflowing the container.

6. Samples are marked for each depth increment in the order 1, 2, 3, etc., collected from the surface downward.

7. Containers are marked occasionally with the location's abbreviation to aid in organization.

8. Place filled containers in a crate or heavy carton. Include a page of accompanying field notes which contain location, surface elevation, total depth drilled, unusual drilling conditions encountered, changes in rock type (see 2.1.2), depth encountered, depth of drill bench with respect to original land surface, thickness of coal seam(s) scheduled for mining, and date sampled. Transport to laboratory.

2.2.3 Sampling From Exploration Cores

2.2.3.1 Principle--

Exploration cores give a vertical column of overburden material. The cores, usually 5 cm (2 in) in diameter, leave intact rock samples. From these cores detailed geologic logging can be accomplished.

2.2.3.2 Comments--

Cores are logged and sampled from the top to the bottom of the core. Hand samples from the drill bench to the surface must be taken by hand if an intact soil core was not obtained.

The geographical location of the core should be located on a U.S.G.S 7 1/2 minute topographic map. Latitude and longitude coordinates are determined to four decimal places using a ruler.

Sample intervals cited are a general rule to follow. Special characteristics of the core will ultimately determine sample interval and number of samples required.

2.2.3.3 Chemicals--

Hydrochloric acid (HCl), 1 part acid to 3 parts water: Dilute 250 ml of concentrated HCl to 1 liter with distilled water.

2.2.3.4 Materials--

1. Rock hammer.

2. Containers with lids, one-quart. NOTE: One-pint containers may be substituted for smaller samples.

3. Felt-tip pen or other marker for legibly labeling sample containers.

4. Crate or heavy corrugated carton for transporting containers to laboratory.

5. Record book.

6. Hand lens, 10 power.

7. Dropper bottle for acid.

2.2.3.5 Procedure (revised and updated from Smith et al., 1974)--

1. Record the following: (a) site location, (b) depth from land surface to top of core, (c) total length of core, (d) elevation of land surface, (e) coal seams scheduled for mining with elevations and thickness, and (f) date sampled.

2. Samples 12 cm (5 in) long are taken from near the center of the represented sample interval.

3. Locate coal seams scheduled for mining.

4. Take a sample representing the total 30 cm (12 in) of material overlying a coal seam scheduled for mining.

5. Take a sample representing the total 30 cm (12 in) of material underlying a coal seam scheduled for mining. NOTE: Samples are not taken of the coal seam scheduled for mining.

6. Determine soil horizons and rock types (see 2.1.2). NOTE: In cores, if the soil horizons are absent, a pit will have to be dug to obtain soil horizon information (see 2.2.4).

7. Take samples. NOTE: Samples are taken from near the center of the represented sample interval. Th sample interval is usually 30 cm (12 in) unless one of the following criteria can be met:

a. Rock members less than 13 cm (5 in) thick are considered transition zones, with the upper half incorporated with the overlying rock member and the lower half incorporated with the underlying rock member. Existence of transition zones should be recorded.

b. When an obvious change in chroma or texture (e.g. high (greater than 3) versus low (less than 2) chroma or coarse versus fine grained sandstone) occurs within a rock type, the two zones are sampled separately.

c. Zones of special interest should be sampled separately regardless of thickness.

d. If a sandstone is determined by an experienced person to have the same characteristics throughout, one sample can represent up to 1.5 meters (5 ft) of a thick-bedded or highly weathered (chroma 3 or higher) sandstone.

e. If the rock type has the same characteristics throughout, as determined by an experienced person, one sample can represent up to 1 meter (3 ft) of carbolith, mudrock, mudstone, shale, limestone, or other rock type.

8. Record sample number, soil horizon or rock type, and sample interval represented by the sample.

9. Record any items of special interest contained in the sample.

10. Place sample in container and label (see 2.2.6).

11. In the laboratory, recheck soil horizons and rock types. Determine soil horizon and rock type color. CAUTION: Above all, use intelligence guided by experience when sampling.

2.2.4 Hand Sampling A Highwall

2.2.4.1 Principle--

A vertical column of samples is needed to represent the different materials contained in the overburden of a coal seam. Samples are taken with a rock hammer from freshly exposed surfaces of the material.

2.2.4.2 Comments--

This procedure should be used only when an exploration core or a blast hole drill is not available or when mining operations expose different strata from those represented by earlier investigations.

Samples must be taken of freshly exposed surfaces. Contact with weathering elements over a long period of time will change the characteristics of exposed surfaces. Samples are taken by hand sampling the highwall at prescribed intervals from the coal to the land surface. One should work along access roads, use an extension ladder, or ropes and cliff climbing techniques to acquire samples. When working near a highwall, remember the presence of loose rock and use care. Hard hats, steel-toed shoes, and other safety equipment are necessities.

If the vertical column of overburden material is inaccessible or incomplete, one can sample the total column by combining lateral movement with vertical sampling, thus establishing a step-like sampling pattern across the highwall.

The geographical location of the sample site should be located on a U.S.G.S. 7 1/2 minute topographic map. Latitude and longitude coordinates are determined to four decimal places by using a ruler.

Sample intervals cited are a general rule to follow. Special characteristics of the highwall will ultimately determine sample interval and number of samples required.

2.2.4.3 Chemicals--

Hydrochloric acid (HCl), 1 part acid to 3 parts water: Dilute 250 ml of concentrated HCl to a volume of 1 liter with distilled water.

2.2.4.4 Materials--

1. Rock hammer and chisel.

2. Extension ladder (if required).

3. Climbing gear (if required).

4. Containers with lids, one-quart capacity, plastic coated cardboard.
NOTE: One-pint containers may be substituted depending on sample size.

5. Felt-tip pen or other marker for legibly labeling sample containers.

6. Crate or heavy corrugated carton.

7. Record book.

8. Hand lens, 10 power.

9. Dropper bottle for acid.

2.2.4.5 Procedure--

1. Record the following: (a) site location, (b) coal seams scheduled for mining with elevations and thicknesses, (c) elevation of original land surface, and (d) date sampled.

2. Samples are taken from near the center of a freshly exposed surface of the sampling interval.

3. Locate coal seams scheduled for mining.

4. Take a sample representing the total 30 cm (12 in) of material overlying a coal seam scheduled for mining.

5. Take a sample representing the total 30 cm (12 in) of material underlying a coal seam scheduled for mining. NOTE: Samples are not taken of the coal seam scheduled for mining.

6. Determine soil horizons and rock types (see 2.1.2).

7. Take samples. NOTE: Samples are taken from near the center of the represented sample interval. The sample interval is usually 30 cm (12 in) unless one of the following criteria can be met:

a. Rock members less than 13 cm (5 in) thick are considered transition zones, with the upper half incorporated with the overlying rock member and the lower half incorporated with the underlying rock member. Existence of transition zones should be recorded.

b. When an obvious change in chroma or texture (e.g. high (greater than 3) versus low (less than 2) chroma or coarse versus fine grained sandstone) occurs within rock type, the two zones are sampled separately.

c. Zones of special interest should be sampled separately regardless of thickness.

d. If a sandstone is determined by an experienced person to have the same characteristics throughout, one sample can represent up to 1.5 meters (5 ft) of a thick bedded or highly weathered (chroma 3 or higher) sandstone.

e. If the rock type has the same characteristics throughout, as determined by an experienced person, one sample can represent up to 1 meter (3 ft) of carbolith, mudrock, mudstone, shale, limestone, or other rock type.

8. For each sample record sample number, soil horizon or rock type, and thickness of material represented by the sample.

9. Record any items of special interest contained in the sample.

10. Place sample in container and label (see 2.2.6).

11. Recalculate depth so that soil horizons and rock types are recorded as depth from land surface.

12. In the laboratory, recheck soil horizons and rock types. Determine soil horizon and rock type color. CAUTION: Above all, use intelligence guided by experience when sampling.

2.2.5 Selective Samples

2.2.5.1 Principle--

Selective samples are taken by hand. They are usually selected from a sampling site on materials of special interest (such as a high pyrite zone, vegetated versus unvegetated area, etc.). These areas of special interest are taken into account in the total site overburden interpretation.

2.2.5.2 Comments--

The type and amount of selective samples acquired depends upon the analyses being performed. Description and sample size varies with the individual sampler and his specific needs or interest.

2.2.5.3 Chemicals--

None required.

2.2.5.4 Materials--

1. Rock hammer.

2. Shovel.

3. Containers with lids, one-quart capacity. NOTE: One-pint containers may be substituted for smaller samples.

4. Felt-tip pen or other marker for legibly labeling sample containers.

5. Record book.

2.2.5.5 Procedure--

1. Use the rock hammer or shovel to acquire sample.

2. Place sample in one-quart container and cover.

3. Label sample (see 2.2.6).

4. Record sample number, site location, surface elevation, date sampled, and reasons for taking sample in the record book.

5. Transport samples to laboratory for analysis.

2.2.6 Labeling Samples

2.2.6.1 Principle--

Samples are labeled to separate one location from another or one sample from another. Four main items are required on each sample label: site location, sample number, depth from original land surface or sample increment, and date sampled.

2.2.6.2 Comments--

Complete information about the site location is necessary. Site location should include mine name (also pit number where available), sample column number (e.g. if more than one column is sampled from the same site) or exploration core number, longitude and latitude, surface elevation, date sampled, and any additional information necessary to locate the sample site.

Sample numbers should be in consecutive order from the top to the bottom of the column. Any samples taken between existing samples should be followed by a letter in alphabetical order or a decimal point and number in numerical order. EXAMPLE: Four samples taken between sample 2 and 3 would be 2A, 2B, 2C, 2D or 2.1, 2.2, 2.3, 2.4.

Depth should be recorded from the original land surface. When original land surface cannot be determined, sample increments (e.g. 30 cm (1 ft), length of rock hammer, etc.) should be recorded. Any change in sample increments should also be recorded.

Any special interest information (such as noticeable gypsum, pyritic zone, etc.) can be added.

2.2.6.3 Chemicals--

None required.

2.2.6.4 Materials--

1. Shovel and/or rock hammer for acquiring samples.

2. Containers with lids, one-quart for holding samples. NOTE: One-pint containers may be substituted depending on sample size.

3. Felt-tip pen or other marker for legibly labeling sample containers.

4. Record book.

2.2.6.5 Procedure--

1. Acquire sample using the sampling procedures (2.2.2, 2.2.3, 2.2.4, or 2.2.5).

2. Place sample in container.

3. Label container with site location, sample number, sample depth or sampling increment, and date sampled.

4. Record data in step 3 in record book if not previously completed during sampling.

2.3 DESCRIBING AND SAMPLING MINESOILS

2.3.1 Describing Minesoil Profiles

2.3.1.1 Principle--

In the new classification system, Soil Taxonomy (USDA, 1975), soils are classified on the basis of characteristics which can be observed or measured in the field and in the laboratory. Such a study requires a vertical cross-section extending from the surface down through 100 cm (40 in) below the surface. Properties of minesoils that often are lacking or different in undisturbed soils include the following:

1. Disordered coarse fragments. If coarse fragments constitute at least 10% of the volume of the control section, they are disordered such that more than 50% will have their long axis at an angle of at least 10% relative to any plane in the profile.

2. Color mottling without regard to depth or spacing in the profile. The mottling involves color differences of at least 2 color chips in the standard Munsell Color Charts. This mottling occurs among fines as well as within coarse fragments or between fines and coarse fragments.

3. Splintery edges on fissile coarse fragments. If coarse fragments are fissile, the edges are frayed or splintery rather than smooth.

4. Bridging voids. Coarse fragments bridging across voids as a result of placement of materials leave discontinuous irregular pores. These pores are larger than those from textural porosity.

5. Thin surface horizon higher in fines. A thin "near-surface horizon" often immediately below a surface pavement of coarse fragments, contains a higher percentage of fines (material less than 2 mm in effective diameter) than any other horizon in the profile to the bottom of the control section.

6. Local pockets of dissimilar material. Local pockets of dissimilar materials, excluding single coarse fragments, may range from 7.6 to 100 cm

in horizontal diameter. These pockets have no lateral continuity and are the result of the original placement of material and not of post-depositional processes.

7. Artifacts. Artifacts (plastics, glass, paper, metal, tires, logs, etc.) may appear in the profile.

8. Carbolithic coarse fragments. Carbolithic coarse fragments are frequently found in non-carbolithic classes of minesoils. These coarse fragments, which are usually associated with a coal horizon, are found in the profile because of moving and mixing of overburden materials.

9. Irregular distribution of oxidizable organic carbon. The irregular distribution of oxidizable carbon with depth in the profile is due to the mixing of overburden materials. Both recent and geologically old carbon compounds are involved.

2.3.1.2 Comments--

It is necessary to sample and describe fresh exposures in soils. Many minesoils present a problem because of a high proportion of coarse fragments. This causes hand digging to be difficult and time consuming. In some cases profile descriptions can be taken from road cuts, gullies, slips, etc. if the exposed surface is scraped or cleaned to remove effects of surface weathering or overwash.

Walk over the area, examining the surface and selecting a site representative of the area in general. Once a site has been selected, an excavation should be made to a depth of at least 100 cm and preferably deeper. The profile should be described to the 100 cm depth. An experienced soil scientist would probably prefer to describe the profile in more detail than will be put in this method. He can do this by following the profile description outlined in the Soil Survey Manual (USDA, 1951, p. 137-141). However, in addition to the information as outlined in the Soil Survey Manual, the properties of Spolents as described in 2.3.1.1 should be noted and recorded.

The following list of morphological features should be noted when describing a minesoil profile:

1. Layers. Minesoils may have different layers which result from placement of materials.

2. Depth. Depth is given in centimeters and is measured from the surface downward (e.g. 0-10 cm).

3. Color of the matrix. See procedure 2.1.3.

4. Mottling. Describe abundance, contrast, size, and color. Since mottling is one of the dominant properties of minesoils, extra care should be taken in describing the mottled patterns. See procedure 2.1.3 for color determination of the mottling.

5. Texture. See procedure 2.1.8.

6. Reaction (pH). If possible, pH should be determined in the field with a pH meter (see procedure 3.2.2). The reaction classes of Spolents are as follows: (a) extremely acid, pH is 4.0 or less, except for carbolithic classes (high-carbon mine waste) which are extremely acid at pH 3.0 or lower; (b) acid, pH is 4.0 to 5.5, except for carbolithic classes which have an acid range from pH 3.0 to 5.5; (c) neutral, pH is 5.5 to 8.0; (d) alkaline, pH is greater than 8.0.

7. Coarse fragments. Total percent by volume of each layer should be estimated in increments of 5% and recorded. Also record percent of each type of coarse fragment, such as shale, sandstone, mudstone, etc. NOTE: It may become necessary to break open the coarse fragments when in doubt.

8. Roots. Record the amount of roots in each layer.

9. Bridging voids. Abundance and size distribution for each layer should be recorded.

10. Artifacts. Record the amount and depth of all artifacts found in the profile. These artifacts can also include buried tree stumps and branches.

11. Pockets. Any pockets of dissimilar material should be described by size, texture, color, and percent abundance.

Anything else that may seem significant or will provide additional information about the profile should be recorded in the field notebook. Some of the miscellaneous things that would be noted are: coatings on coarse fragments, earthworm channels and excretions, concretions, etc.

2.3.1.3 Chemicals--

1. Hydrochloric acid (HCl), 1 part acid to 3 parts water: Dilute 250 ml of concentrated HCl to a volume of 1 liter with distilled water.

2. Water (H_2O).

2.3.1.4 Materials--

1. Spud bar.

2. Long handled shovel.

3. Marking knives.

4. Diapers or cloth towel.

5. Field notebook.

6. Portable pH meter or pH kit.

7. Munsell Soil Color Charts.

8. Tile spade.

9. Plastic sample bags, 20 pound capacity.

10. Felt-tip pen or other marker for legibly labeling sample bags.

2.3.1.5 Procedure--

1. Select the area on the minesoil to be described. Record location, surface elevation, and description of area where pit is to be dug in the field notebook.

2. Dig a pit vertically downward from the surface through a depth of at least 100 cm.

3. After the pit has been dug, clean the loose material from the face of the pit with a knife.

4. Study the features of the freshly exposed face of the pit.

5. Put a marking knife at the bottom of layer one. NOTE: This is the surface layer which will be higher in fines than the rest of the profile.

6. After layer one has been marked, study the rest of the exposed profile to see if any noticeable changes in material occur (e.g. a layer of carbolithic material sandwiched between layers of high chroma sandstone). In other words, there must be something which visually separates the material below layer one into different layers.

7. If there are no visual differences in the material from the bottom of layer one to the 100 cm depth, then place a knife at 25 cm intervals from the knife that marks the bottom of layer one.

8. Number the layers going downward: 1, 2, 3, etc.

9. Describe all the properties that have been put forth in 2.3.1.1 and 2.3.1.2 for each layer.

10. Record all descriptive material for each layer in the field notebook.

11. Sample each layer of the profile according to procedure 2.3.2.

12. Label all samples as to layer number, area, location (longitude, latitude, surface elevation), and transport back to laboratory (see 2.2.6).

2.3.2 Sampling Minesoils for Classification and Fertility

2.3.2.1 Principle--

An important source of error when investigating a soil body is taking samples for detailed laboratory measurements. Laboratory analyses only measure parameters that are contained in a particular soil sample. If the soil sample is taken haphazardly and is not representative of the soil body from which it was taken, then laboratory measurements are meaningless.

Sampling of minesoils should be done for a particular purpose. In minesoils (very young soils in pedogenic development) a measure of the variability within the soil body is extremely useful. Therefore, subdivisions of the minesoil, based on visual differences, are sampled and analyzed.

2.3.2.2 Comments--

The size of the sample depends on the purposes for sampling. For example, several small samples should be taken if only pH is to be measured. On the other hand, large samples are needed for doing all the measurements in section 3.

It is extremely important to label all samples correctly. Also, all sample numbers and descriptive information for each sample should be recorded in the record book along with a sketched map of the minesoil showing sampling subdivisions.

2.3.2.3 Chemicals--

None required.

2.3.2.4 Materials--

1. Record book.
2. Felt-tip pen or other marker for legibly labeling sample bags.
3. Spud bar.
4. Long handled shovel.
5. Tile spade.
6. Paddy shovel (D-handled dirt shovel).
7. Plastic bags, 20 lb capacity.
8. Plastic bags, sandwich size.
9. Rock hammer, flat nosed.
10. Knife, or large spatula.
11. Sieve, 7.6 cm (3 in) openings.

2.3.2.5 Procedure--

1. Walk or ride over the minesoil and note all visual differences such as color, texture, surface elevation, erosion, volunteer vegetation, and wet spots.

2. Draw a map of the minesoil dividing the surface area into different sampling units. NOTE: These subdivisions are made on the basis of visual differences as noted in step 1.

3. After the map of the area has been drawn and all sampling subdivisions have been made, examine the area once more to be sure that everything has been taken into account. Record longitude, latitude, and surface elevation of sampling area.

4. Do either 2.3.2.5.1 or 2.3.2.5.2.

2.3.2.5.1 Profile Sampling--

1. After deciding where the profile will be placed, take the spud bar and long handled shovel and dig a pit vertically from the surface to a depth of 100 cm.

2. After the pit has been dug so that a vertical cross section of the minesoil is exposed, describe the minesoil according to method 2.3.1.

3. Take a sample from each layer that has been described according to 2.3.1. Also, any major variations within a layer, such as pockets of dissimilar material that are of special interest, should be sampled.

4. All samples should be labeled as to pit number, layer number, depth, etc. NOTE: The more information gathered about a particular sample, the more useful the sample.

5. Pass all samples for a particular layer through a sieve with 7.6 cm openings and into a large plastic bag. Discard all material caught on the sieve after visually estimating the percentage of the total sample retained on the sieve. This information should be recorded and included in the final interpretation.

6. After sample has been put into labeled plastic bag, tie bag with twine and transport to laboratory.

2.3.2.5.2 Surface Sampling--

1. After the area has been subdivided into different units, determine the number of samples required per subdivision. NOTE: At least one sample per subdivision should be taken; however, depending on what analyses and what variability may be contained in one subdivision, more samples can be taken. Three samples per subdivision are preferred. If only pH is to be tested, take as many small samples (sandwich baggies full of material) as possible per each subdivision.

2. In each subdivision take representative samples of the 0 to 7.6 cm depth with a paddy shovel. NOTE: If a minesoil has been "topsoiled" with material that can be worked with farm implements, the samples are taken from the surface to a depth of 16 cm.

3. Pour sample from paddy shovel into a plastic ice bag through a sieve with 7.6 cm openings until bag is three-fourths full. NOTE: A visual estimate of the percentage of total sample retained on the sieve should be recorded and included in the ultimate interpretation. The material retained on the sieve is then discarded.

4. Label the sample bag carefully recording as much information about the site as possible and date sampled. Also record this information in the record book.

5. Transport samples back to laboratory.

SECTION 3

SAMPLE PROCESSING AND LABORATORY ANALYSES

3.1 CHARACTERIZING, SUBSAMPLING AND CRUSHING SAMPLES

3.1.1 Characterizing Samples

3.1.1.1 Principle--

Even when careful measures are taken, errors may result in the labeling and collecting of samples in the field. This procedure is a laboratory check list to verify that all information was gathered when samples were collected.

3.1.1.2 Comments--

Samples should be checked for errors as soon as possible after sampling. The time spent rechecking field data and samples may eliminate missing data, errors in data, or unnecessary trips back to the sample site to collect missing information.

3.1.1.3 Chemicals--

None required.

3.1.1.4 Materials--

1. Field record book.

2. Collected samples.

3.1.1.5 Procedure--

1. Verify the following:

a. Sample site has been located correctly on a topographic map and longitude and latitude have been recorded.

b. Surface elevation at sampling site has been recorded.

c. Name, depth, and thickness of coal seam(s) scheduled for mining have been recorded.

d. Total overburden thickness has been recorded.

e. For each sample the correct site location, sample number, soil or rock type (if done in field), sampling interval represented by sample, depth from surface, and date sampled have been recorded in the record book. NOTE: Both the interval represented by the sample and the depth from the surface should be recorded even though one can be calculated from the other.

2. For each overburden column, check to make sure that each sample container has a sample, is numbered correctly, and is labeled correctly.

3. Determine rock type in the laboratory if not completed in the field.

4. Determine color in the laboratory on ground (less than 60 mesh) sample.

3.1.2 Subsampling and Grinding Rock and Native Soil Samples

3.1.2.1 Principle--

Crushing reduces the field sample into a convenient size range for use with various laboratory analyses. Samples are crushed, subsampled, and ground to pass a 0.25 mm (60 mesh) sieve.

3.1.2.2 Comments--

Crusher and pulverizer should be cleaned after each sample to avoid contamination between samples.

Native soils are soil horizons which are taken above and are treated as an extension of a core, blast hole, or hand sampled highwall column. Soil and rock samples should be air dried, not oven dried, before subsampling and grinding.

3.1.2.3 Chemicals--

None required.

3.1.2.4 Materials--

1. Crusher, chipmunk, motor driven, capable of crushing samples to less than 6.35 mm (0.25 in) (Cat. No. 5-60836, Sargent-Welch Scientific Company; or equivalent).

2. Pulverizer, capable of crushing samples to less than 60 mesh (Fen Corp., Wickliffe, Ohio, Model PA-M; or equivalent).

3. Mortar and pestle, cast iron (Cat. No. 12-976, Fisher Scientific Company; or equivalent).

4. Sieve, 0.25 mm openings (60 mesh).

5. Sieve, 6.35 mm (0.25 in) openings (optional).

6. Vials, plastic with snap caps, 148 cc (40 drams) capacity.

7. Container, plastic or waxed paper, 1 liter (32 oz) capacity.

3.1.2.5 Procedure (revised and updated from Smith et al., 1974)--

1. Spread sample evenly on a sheet of brown paper and allow to air dry. NOTE: Sample may have to be mixed periodically to speed drying.

2. After drying, the field sample is split into two representative subsamples. One subsample is placed in a container, labeled, and stored for physical analyses or individual preference tests.

3. The other subsample is crushed to 6.35 mm (0.25 in) or smaller with a Chipmunk crusher. If a crusher is not available, the material can be crushed using a hammer or mortar and pestle until it passes through a sieve with 6.35 mm openings. NOTE: This step may be omitted on most native soil samples.

4. Place sample in 1 liter container and cover. NOTE: Containers should not be more than two-thirds full or mixing (step 5) will be impaired.

5. Tumble container end-over-end until material is thoroughly mixed.

6. Place three heaping teaspoons of the mixed material in the pulverizer. Material is pulverized until it passes a 0.25 mm (60 mesh) sieve. NOTE: A cast iron mortar and pestle can be substituted for the pulverizer.

7. Place pulverized material in plastic vial for laboratory use.

8. Label vial with the sample identification shown on the field container.

9. Mix sample thoroughly by tumbling the vial end-over-end before subsampling for laboratory procedures (primarily chemical analyses).

3.1.3 Subsampling and Grinding Minesoil Samples

3.1.3.1 Principle--

See 3.1.2.1

3.1.3.2 Comments--

Samples should be air dried before processing begins. Samples should never be oven dried before processing.

Pulverizer should be cleaned after each sample to avoid contamination between samples.

3.1.3.3 Chemicals--

None required.

3.1.3.4 Materials--

1. Wooden rolling pin (kitchen style).

2. Pulverizer, capable of crushing samples to less than 60 mesh (Fen. Corp., Wickliffe, Ohio, Model PA-M; or equivalent).

3. Sieve, 20 cm (8 in) diameter, 19 mm (0.75 in) openings.

4. Sieve, 20 cm (8 in) diameter, 6.35 mm (0.25 in) openings.

5. Heavy brown kraft paper.

6. Vials, plastic with snap caps, 148 cc (40 drams) capacity.

7. Containers, large enough to contain sample fractions.

8. Large spatula.

3.1.3.5 Procedure--

1. Pour field sample out onto a large square of brown paper. Spread material evenly and allow to air dry. NOTE: Sample may have to be mixed periodically to speed drying.

2. After drying, the field sample is split into two representative subsamples. One subsample is placed in a container, labeled, and stored for physical analyses or individual preference tests.

3. The other subsample is placed between two sheets of brown paper and crushed by moderately rolling over the top sheet with a rolling pin. This process is continued until the entire field sample has been processed. NOTE: Do not allow paper fragments to become incorporated with the soil sample. Do not crush rock fragments.

4. Pass the crushed material through a sieve with 19 mm openings and discard material retained on the sieve.

5. All material passing the 19 mm sieve is crushed to pass through a sieve with 6.35 mm openings.

6. Place sieved sample in a 1 liter container and cover. NOTE: Container should not be more than two-thirds full or mixing (step 7) will be impaired.

7. Tumble container end-over-end until material is thoroughly mixed.

8. Place three heaping teaspoons of the mixed material in the pulverizer. Material is pulverized until it passes a 0.25 mm (60 mesh) sieve. NOTE: A cast iron mortar and pestle can be substituted for the pulverizer.

9. Place pulverized material in a plastic vial for laboratory use.

10. Label vial with the sample identification shown on the field container.

11. Mix sample thoroughly by tumbling the vial end-over-end before subsampling for laboratory procedures (primarily chemical analyses).

3.2 CHEMICAL METHODS

3.2.1 Summary

Chemical methods for characterizing overburdens and minesoils are given. For a particular parameter, more than one method may be listed. This will allow the user of the manual some freedom of choice.

The determination of toxic or nontoxic materials due to acidity is overriding in importance in the Appalachian and Eastern and Western Interior Coal Provinces. The methods for determining toxic or potentially toxic materials are given high priority and are listed at the very front of the chapter. Methods 3.2.2, 3.2.3, 3.2.4, and 3.2.6 are used to determine the acid-base balance of minesoils and overburdens.

Next in importance is the nutrient status of the overburden materials. Nutrient status can be measured by using methods 3.2.5, 3.2.6, and 3.2.15. These methods give a measure of plant nutrients such as phosphorus, potassium, calcium, magnesium, and nitrogen. A knowledge of what plant nutrients are contained in an overburden material enables the mine operator to efficiently plan the mining operation so that full advantage can be taken of these nutrients in the resulting minesoil.

For more intensive study of minesoils and overburden materials, procedures for determining the cation exchange capacity (3.2.16 and 3.2.17) are given. Ways of estimating the lime requirement in minesols are presented in methods 3.2.7 through 3.2.10. Also, methods applicable to arid and semiarid regions have been included.

3.2.2 Paste pH

3.2.2.1 Principle--

Perhaps the most commonly measured soil characteristic is pH. Soil pH was defined by Sorensen (1909) as the negative logarithm of the hydrogen-ion concentration. However, in actuality, hydrogen-ion activity is measured instead of hydrogen-ion concentration.

Soil pH is measured by a glass electrode incorporated with a pH meter for this procedure. Water is added to the sample forming a paste. The electrode is placed in the paste with pH being read directly from the meter.

3.2.2.2 Comments--

Six factors affecting the measurement of pH are: (1) drying the soil sample during preparation; (2) soil:water ratio used; (3) soluble salts content;

(4) seasonally influenced carbon dioxide content; (5) amount of grinding given the soil; and (6) electrode junction potential (Jackson, 1958; Peech, 1965).

Care must be taken to insure electrode life and accurate pH measurements: (1) Electrode should not remain in the sample longer than necessary for a reading, especially if more alkaline than pH 9.0. (2) Electrode should be washed with a jet of distilled water from a wash bottle after every measurement (sample or buffer solution). (3) Electrode should be dipped in dilute (1 part acid to 3 parts water) hydrochloric acid for a few seconds and washed with distilled water to remove any calcium carbonate film which may form, especially from alkaline samples. (4) Drying out of the electrode should be avoided. Electrode is cleaned and suspended in distilled water (which is protected from evaporation) for storage. (6) Place pH meter in standby position when electrode is not in a solution (Jackson, 1958; Peech, 1965).

The pH meter and electrode should be standardized with buffers differing by 3 or 4 pH units, such as 4.0 and 7.0, before beginning a series of measurements. After every tenth measurement, recheck the standardization with both buffers. Care should be taken not to contaminate one buffer with the other buffer or with the test solution. Never return used standard buffers to their stock bottles. The procedure describes the technique for measuring pH with a glass electrode and meter. If pH is taken in the field using color paper strips or indicator solutions, modification will have to be made by qualified personnel to the procedure.

3.2.2.3 Chemicals--

1. Standard buffer solutions, pH 4.00 and pH 7.00.

2. Distilled water (H_2O).

3.2.2.4 Materials--

1. pH meter (Corning model 12 or equivalent) equipped with combination electrode.

2. Paper cups, 30 ml (1 oz) capacity.

3. Plastic cups.

4. Stirring rod.

5. Wash bottle containing distilled water.

6. Balance, can be read to 0.1 g.

3.2.2.5 Procedure--

1. Turn on, adjust temperature setting, and "zero" pH meter per instruction manual.

2. Place pH 4.0 and pH 7.0 standard buffers in two plastic cups (one buffer in each cup). NOTE: NEVER return used buffers to stock bottles.

3. Place electrode in the pH 7.0 buffer.

4. Adjust pH meter to read pH 7.0.

5. Remove electrode from buffer solution and wash with a jet of distilled water from a wash bottle.

6. Place electrode in the pH 4.0 buffer and check the pH reading. NOTE: If pH meter varies more than ± 0.1 pH units from 4.0, something is wrong with the pH meter, electrode, or buffers.

7. Weigh 10 g of less than 60 mesh material into a paper cup.

8. Add 5 ml of distilled water to sample. NOTE: Do not stir! Allow water to wet sample by capillary action without stirring. With most overburden and minesoils materials, the 2:1 (soil:water) ratio provides a satisfactory paste for pH measurements; however, for the very coarse textured and the very fine textured material, more material or water can be added to bring the soil near saturation. At near saturation conditions, water should not be puddled nor dry soil appear at the surface.

9. Stir sample with a spatula until a thin paste is formed adding more water or soil as required to keep soil at saturation point. NOTE: At saturation, the soil paste glistens as it reflects light and the mixture slides off the spatula easily. Wash the spatula with a jet of distilled water before stirring another sample.

10. Place electrode in paste and move carefully about to insure removal of water film around the electrode. CAUTION: Do not trap particles between electrode and inside surface of the sample container. Electrodes are easily scratched. Contact between paste and electrode should be gentle to avoid both impact and scratching damage, especially in sandy samples.

11. When reading remains constant, record pH and remove electrode from paste. Carefully wash electrode with distilled water to insure removal of all paste. If all pH measurements are completed, the electrode should be stored in a beaker of distilled water. NOTE: After every 10 samples, check meter calibration with standard buffers.

3.2.3 Neutralization Potential

3.2.3.1 Principles--

The amount of neutralizing bases, including carbonates, present in overburden materials is found by treating a sample with a known excess of standardized hydrochloric acid. The sample and acid are heated to insure that the reaction between the acid and the neutralizers goes to completion.

The calcium carbonate equivalent of the sample is obtained by determining the amount of unconsumed acid by titration with standardized sodium hydroxide (Jackson, 1958).

3.2.3.2 Comments--

A fizz rating of the neutralization potential is made for each sample to insure the addition of sufficient acid to react all the calcium carbonate present.

During digestion, do not boil samples. If boiling occurs, discard sample and rerun. Before titrating with acid, fill buret with acid and drain completely. Before titrating with base, fill buret with base and drain completely to assure that free titrant is being added to the sample.

3.2.3.3 Chemicals--

1. Carbon dioxide-free water: Heat distilled water just to boiling in a beaker. Allow to cool slightly and pour into a container equipped with ascarite tube. Cool to room temperature before using.

2. Hydrochloric acid (HCl) solution, 0.1 N, certified grade (Fisher So-A-54 or equivalent).

3. Sodium hydroxide (NaOH), approximately 0.5 N: Dissolve 20.0 g of NaOH pellets in carbon dioxide-free water and dilute to 1 liter. Protect from CO_2 in the air with ascarite tube. Standardize solution by placing 50 ml of certified 0.1 N HCl in a beaker and titrating with the prepared 0.5 N NaOH until a pH of 7.00 is obtained. Calculate the Normality of the NaOH using the following equation:

$$N_2 = (N_1V_1)/V_2, \text{ where:}$$

V_1 = Volume of HCl used.

N_1 = Normality of HCl used.

V_2 = Volume of NaOH used.

N_2 = Calculated Normality of NaOH.

4. Sodium hydroxide (NaOH) approximately 0.1 N: Dilute 200 ml of 0.5 N NaOH with carbon dioxide-free water to a volume of 1 liter. Protect from CO_2 in air with ascarite tube. Standardize solution by placing 20 ml of certified 0.1 N HCl in a beaker and titrating with the prepared 0.1 N NaOH until a pH of 7.00 is obtained. Calculate the Normality of the NaOH using the equation in 3.2.3.3 No. 3.

5. Hydrochloric acid (HCl), approximately 0.5 N: Dilute 42 ml of concentrated HCl to a volume of 1 liter with distilled water. Standardize solution by placing 20 ml of the known Normality NaOH prepared in 3.2.3.3 No. 3 in a beaker and titrating with the prepared HCl until a pH of 7.00 is obtained.

Calculate the Normality of the HCl using the following equation:

$$N_1 = (N_2V_2)/V_1, \text{ where:}$$

V_2 = Volume of NaOH used.

N_2 = Normality of NaOH used.

V_1 = Volume of HCl used.

N_1 = Calculated Normality of HCl.

6. Hydrochloric acid (HCl), approximately 0.1 N: Dilute 200 ml of 0.5 N HCl to a volume of 1 liter with distilled water. Standardize solution as in 3.2.3.3.5, but use 20 ml of the known Normality NaOH prepared in 3.2.3.3 No. 4.

7. Hydrochloric acid (HCl), 1 part acid to 3 parts water: Dilute 250 ml of concentrated HCl with 750 ml of distilled water.

3.2.3.4 Materials--

1. Flasks, Erlenmeyer, 250 ml.

2. Buret, 100 ml (one required for each acid and one for each base).

3. Hotplate, steam bath can be substituted.

4. pH meter (Corning Model 12 or equivalent) equipped with combination electrode.

5. Balance, can be read to 0.01 g.

3.2.3.5 Procedure (revised and updated from Smith et al., 1974)--

1. Place approximately 0.5 g of sample (less than 60 mesh) on a piece of aluminum foil.

2. Add one or two drops of 1:3 HCl to the sample. The presence of $CaCO_3$ is indicated by a bubbling or audible "fizz."

3. Rate the bubbling or "fizz" in step 2 as indicated in Table 1.

4. Weigh 2.00 g of sample (less than 60 mesh) into a 250 ml Erlenmeyer flask.

5. Carefully add HCl indicated by Table 1 into the flask containing sample.

6. Heat nearly to boiling, swirling flask every 5 minutes, until reaction is complete. NOTE: Reaction is complete when no gas evolution is visible and particles settle evenly over the bottom of the flask.

TABLE 1. VOLUME AND NORMALITY OF HYDROCHLORIC ACID USED FOR EACH FIZZ RATING

	HCl	
Fizz Rating	(ml)	(Normality)
None	20	0.1
Slight	40	0.1
Moderate	40	0.5
Strong	80	0.5

7. Add distilled water to make a total volume of 125 ml.

8. Boil contents of flask for one minute and cool to slightly above room temperature. Cover tightly and cool to room temperature. CAUTION: Do not place rubber stopper in hot flask as it may implode upon cooling.

9. Titrate using 0.1 N NaOH or 0.5 N NaOH (concentration exactly known), to pH 7.0 using an electrometric pH meter and buret. The concentration of NaOH used in the titration should correspond to the concentration of the HCl used in step 5. NOTE: Titrate with NaOH until a constant reading of pH 7.0 remains for at least 30 seconds.

10. If less than 3 ml of the NaOH is required to obtain a pH of 7.0, it is likely that the HCl added was not sufficient to neutralize all of the base present in the 2.00 g sample. A duplicate sample should be run using the next higher volume or concentration of acid as indicated in Table 1.

11. Run a blank for each volume or normality of acid using steps 5,7, 8, and 9.

3.2.3.6 Calculations--

1. Constant (C) = (ml acid in blank)/(ml base in blank).

2. ml acid consumed = (ml acid added) - (ml base added X C).

3. Tons $CaCO_3$ equivalent/thousand tons of material = (ml of acid consumed) X (25.0) X (N of acid).

3.2.4 Maximum Potential Acidity by Total Sulfur Determination

3.2.4.1 Principles--

This method measures the total sulfur in a sample. If all of the total sulfur occurs in pyritic forms, the calculation of maximum potential acidity from sulfur corresponds with actual potential acidity from sulfur. But if part of the sulfur occurs in other forms, the maximum as calculated will be too high. This is the reason that such calculations are referred to as maximums and in doubtful cases approximate determinations should be made which rule out other sulfur forms (see 3.2.6). These determinations are not necessary when the maximum acid from total sulfur is within safe limits.

A sample is heated to approximately 1600°C. A stream of oxygen is passed through the sample during the heating period. Sulfur dioxide is released from the sample and collected in a dilute hydrochloric acid solution containing potassium iodide, starch, and a small amount of potassium iodate. This solution is automatically titrated with a standard potassium iodate solution.

A trace amount of potassium iodate reacts with potassium iodide and dilute hydrochloric acid to yield free iodine, potassium chloride and water. The free iodine combines with the sulfur dioxide and water to yield sulfuric acid and hydroiodic acid. The amount of potassium iodate solution used during the titration is recorded. The calculation of the percent total sulfur is based on the potassium iodate measurement (Smith et al., 1974).

3.2.4.2 Comments--

Some samples, e.g. coal, when first placed in the furnace may change the color of the solution in the titration vessel to pink or purple (probably due to organic compounds). Some samples may contain halogens (iodine, chlorine, fluorine) which darken the solution in the titration vessel and will therefore produce results that are low. The halogen problem, if encountered, may be eliminated by the use of an antimony trap between the furnace and titration assembly. Interference may result with samples high in nitrogen; however, this does not appear to happen with rock samples. Additional information can be obtained by reading Leco Equipment Application 120 and Instructions for Analysis of Sulfur in Hydrocarbons by the Leco High Frequency Combustion Titration Procedure.

Materials with a low chroma (2 or less) may have a high (over 1.0%) sulfur content; therefore, use a 0.250 g sample when the chroma of the material is 1 or 2. If the chroma of the material is zero, a 0.100 g sample is used. If sulfur is not detectable or more accurate values are desired in this sample size, increase to next highest sample size and rerun.

Read entire manuals on both the Leco Induction Furnace and the Automatic Titrator.

Periodically clean titration chamber and associated glassware with acetone or concentrated hydrochloric acid and rinse thoroughly with distilled water.

The following procedure is for use with a LECO Induction Furnace, Model 521 with Automatic Sulfur Titrator, Model 532. Other similar or advanced models of this instrumentation may perform equally well; however, the following procedure will require detailed modifications by a qualified person for application to other instruments.

3.2.4.3 Chemicals--

1. Iron chip accelerator (Leco number 501-077).

2. Iron powder accelerator (Leco number 501-078).

3. Copper ring (Leco number 550-189).

4. Magnesium oxide (MgO).

5. Potassium iodate (KIO_3), 0.0052 N: Dissolve 1.110 g KIO_3 in distilled water and dilute to 1 liter.

6. Hydrochloric acid (HCl) solution: Dilute 15 ml of concentrated HCl to a volume of 1 liter with distilled water.

7. Arrowroot starch solution: Dissolve 4.0 g of arrowroot starch (Leco number 501-061) in 100 ml of distilled water in a 250 ml beaker. Stir on a mechanical stirrer with a stirring bar. While starch is stirring, boil 300 ml of distilled and deionized water in a 600 ml beaker. Remove from heat when boiling point is reached. Remove starch from stirrer. Place boiled water on mechanical stirrer with stirring bar. While water is continually stirring, add 5 ml of starch mixture in 20 second intervals until all starch solution has been added. Place a small amount of the solution in the 600 ml beaker back into the 250 ml beaker that contained the starch mixture. Wash beaker by hard swirling and then pour contents back into the 600 ml beaker. Continue stirring solution in the 600 ml beaker allowing solution to cool to 40°C. Add 12.0 g of potassium iodide (KI). Continue stirring for 15 to 20 minutes.

8. Potassium iodide (KI).

9. Sulfur standards (Leco number 501-502).

3.2.4.4 Materials--

1. Leco Automatic Sulfur Analyzer, package unit, number 634-700.

2. Scoops, 0.2 ml volume.

3. Ceramic crucibles with porous covers.

4. Carboys, 19 liters (5 gal).

5. Tongs.

6. Glass wool.

7. Oxygen regulators.

8. Mechanical stirrer.

9. Stirring bar.

10. Combustion tube, hydrocarbon (Leco number 519-004).

11. Hot plate.

12. Balance, can be read to 0.001 g.

3.2.4.5 Procedure (revised and updated from Smith et al., 1974)--

NOTE: Read entire manuals on Leco Furnace, Automatic Titrator and this entire procedure before starting.

1. Place one level scoop of iron chips in crucible.

2. Weigh 0.500 g of sample (less than 60 mesh) into the crucible.

NOTE: For samples that are suspected to contain over 1% sulfur or have a chroma of less than 2, see 3.2.4.2.

3. Add one scoop MgO.

4. Add one copper ring and then one scoop of iron powder.

5. Gently shake the crucible to evenly cover the bottom and place one porous cover on the crucible.

6. Turn on "Filament Voltage" grid tap to medium position.

7. Wait for one minute then turn "High Voltage" switch to ON.

8. Set "Titrate-Endpoint" switch to its middle position.

9. Turn on titrator (upper left switch above "Endpoint Adjust").

10. Drain "Titration Vessel" completely.

11. Set timer switch to ON, adjust timer to 10 minutes, or a time sufficient to satisfy steps 25, 26, and 27.

12. Slosh carboys containing HCl and KIO_3 to mix the condensate on the walls of the container.

13. Fill "Iodate Buret."

14. Fill "Titration Vessel" approximately one-third full with the HCl solution.

15. Turn on oxygen. Set the pressure to 15 psi, and the flow rate to 1.0 liter per minute. NOTE: Oxygen flow must be started before starch is added.

16. Raise the "Locking Mechanism Handle" WITHOUT a sample crucible on the pedestal, and lock in place. NOTE: Make sure there is an airtight contact between sample platform and combustion chamber by observing a vigorous bubbling in the "Titration Vessel" chamber.

17. Add one measure (5 ml) of starch solution. NOTE: If solution in "Titration Vessel" chamber turns turbid or yellow after starch solution is added, turn off the instrument following steps 33 through 39 and make NEW starch solution.

18. Set "Titrate-Endpoint" switch to "Endpoint."

19. After a few seconds when titrant level in "Iodate Buret" has stopped falling (Buret reading should be no more than 0.004) the solution in the "Titration Vessel" chamber should be a deep blue. NOTE: If the solution is a pale blue or almost black, turn off the instrument following steps 33 through 39 and make NEW starch solution.

20. Set "Titrate-Endpoint" switch to middle position and lower "Locking Mechanism Handle."

21. Refill "Iodate Buret."

22. Place sample crucible on pedestal, making sure it is centered, and carefully raise "Locking Mechanism Handle" and lock in place.

NOTE: Make sure there is an airtight contact between sample platform and combustion chamber by observing a vigorous bubbling in the "Titration Vessel" chamber.

23. Set "Titrate-Endpoint" switch to Titrate, or if it is known that sample will evolve SO_2 slowly, set switch at Endpoint. The Endpoint setting acts as a "Fine Control" allowing buret valve to discriminate smaller increments.

24. Push RED button on timer to start analysis.

25. Plate current must go to 400-450 ma for at least 2 minutes during the analysis; if not, reweigh and rerun sample.

26. Adjust rheostat to prevent plate current from exceeding 450 ma.

27. When buret reading does not change for 2 minutes, and Plate Current has achieved 400 to 450 ma, it can be assumed that all of the sulfur has been removed from the sample. If buret reading is still changing when timer shuts off instrument, set Timer Switch to OFF, which restarts furnace, leave furnace on until buret is stable for 2 minutes, then turn Timer Switch to ON.

28. Set "Titrate-Endpoint" to middle position. IMPORTANT: Record titration reading.

29. Lower sample platform, remove crucible using tongs, place fresh sample crucible in place, but do not close sample chamber.

NOTE: Slightly drain titrating chamber to maintain original level. Drain, flush, and refill titrating chamber every 3rd sample, or more often if a large quantity of titrant was used by the previous sample (steps 16-22).

30. Refill KIO_3 buret.

31. Close sample chamber, making sure it is tight. Check endpoint (steps 18, 19 and 21).

32. Go to step 23 and continue until all samples have been processed.

33. Turn "Titrate-Endpoint" switch to mid position.

34. Turn off main O_2 valve on top of tank.

35. Turn off "High Voltage."

36. Turn off Automatic Titrator.

37. Drain titration chamber; flush twice with a chamber full of HCl solution or water, cover and leave chamber full of HCl solution.

38. If O_2 has stopped bubbling in the purifying train, turn off small knurled valve on gauge outlet.

39. Turn off "Filament Voltage."

3.2.4.6 Calculations--

1. Percent sulfur. NOTE: Percent sulfur is dependent upon the concentration of potassium iodate titrant and sample size.

A. Using 1.110 g KIO_3/L and 0.500 g sample (0.005 - 1.00% sulfur range)
%S = Buret reading X 5.0.

B. Using 1.110 g KIO_3/L and 0.250 g sample (0.010 - 2.00% sulfur range)
%S = Buret reading X 10.0.

C. Using 1.110 g KIO_3/L and 0.100 g sample (0.025 - 5.00% sulfur range)
%S = Buret reading X 25.0.

2. To convert % sulfur to maximum $CaCO_3$ equivalents: Multiply % sulfur by 31.25 to get tons $CaCO_3$ equivalent/1000 tons of material.

3.2.5 Sodium Bicarbonate Extractable Phosphorus

3.2.5.1 Principle--

This method is a non-destructive extraction of phosphorus from the surfaces of particles. The pH of extracting solution remains nearly constant during the extraction procedure.

The concentration of phosphorous in solution increases in calcareous, alkaline or neutral soils containing calcium phosphates since the concentration of calcium decreases due to the precipitation of calcium as calcium carbonate. In the presence of the solid-phase calcite, the concentration of calcium is 6×10^{-7} M in the extracting solution at equilibrium. As the pH rises, the phosphorous concentration increases in acid soils containing aluminum and iron phosphates. Secondary precipitation reactions are reduced to a minimum in acid and calcareous soils because the aluminum, calcium, and iron concentrations remain at a low level in this extractant (Olsen and Dean, 1965).

3.2.5.2 Comments--

Temperature of the extacting solution and the shaking speed may cause variations in the results. Phosphorous increases approximately 0.43 ppm for each degree rise in temperature between 20° and 30°C for soils testing between 5 and 40 ppm of phosphorous.

Plastic containers should be used to store the extracting solution. If glass is used, a fresh solution should be prepared every month, since the pH tends to increase with time resulting in a higher value for extractable phosphorous.

This method is especially important for overburdens or minesoil because carbonates often are present, even when the paste pH is below 7.

A shaking speed of 2 should be used on the Burrell wrist-action shaker. Other shakers may be used, but when the speed increases greatly from that of the Burrell shaker, somewhat higher results may be obtained.

3.2.5.3 Chemicals--

1. Sodium bicarbonate ($NaHCO_3$), 0.5 M: Dissolve 42.0 g of $NaHCO_3$ and dilute to 1 liter with carbon dioxide-free water (see 3.2.3.3 No. 1). Adjust to pH 8.5 with 1 N NaOH. Protect from CO_2 in air with soda lime or ascarite in a guard tube. Store in polyethylene container and make fresh every 2 months.

2. Ammonium molybdate ($(NH_4)_6Mo_7O_{24} \cdot 4H_2O$): Dissolve 15.0 g of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 300 ml of warm distilled water. Filter if cloudy and allow to cool. Gradually add 342 ml of 12.0 M (37%) HCl. Dilute to 1 liter with distilled and deionized water.

3. Stannous chloride ($SnCl_2 \cdot 2H_2O$), stock solution: Dissolve 10.0 g of $SnCl_2 \cdot 2H_2O$ (large crystals) in 25 ml of 12.0 M (37%) HCl. Store in brown glass bottle in a refrigerator. Prepare fresh every 2 months.

4. Stannous chloride ($SnCl_2 \cdot 2H_2O$), dilute solution: Mix 0.5 ml of $SnCl_2 \cdot 2H_2O$ stock solution with 66 ml of distilled water. Prepare dilute solution for each set of determinations.

5. Potassium phosphate (KH_2PO_4), standard phosphorus stock solution: Dissolve 0.4393 g of KH_2PO_4 with 500 ml of distilled water and dilute to 1 liter. Add 5 drops of toluene to reduce microbial growth. This is a 100 ppm P standard.

6. Potassium phosphate (KH_2PO_4), dilute solution: Dilute 20 ml of KH_2PO_4 stock solution to 1 liter with distilled water. NOTE: This solution contains 2 micrograms of P per ml (2 ppm).

7. Toluene ($C_6H_5CH_3$).

8. Hydrochloric acid (HCl), 12 M (37%).

9. Decolorizing charcoal, Darco G-60 (J. T. Baker Co. or equivalent).

3.2.5.4 Materials--

1. Flasks, Erlenmeyer, 50 ml with stoppers.

2. Flasks, volumetric, 25 ml, with caps.

3. Flask, volumetric, 1000 ml.

4. Funnels, 60 mm diameter.

5. Funnel rack.

6. Beakers, 50 ml.

7. Cylinder, graduated 25 ml.

8. Pipet, 20 ml.

9. Pipet, 10 ml.

10. Pipet, 5 ml.

11. Pipet, 1 ml.

12. Filter paper, 110 mm diameter, medium porosity, ashless (Whatman 40, S & S 589, or equivalent).

13. Balance, can be read to 0.0001 g.

14. Shaking machine, Wrist-Action (Burrell model BB or equivalent).

15. Colorimeter or Spectrophotometer, with filter or adjustment to provide 660 nm incident light.

16. Cuvettes or matched test tubes to fit above colorimeter.

17. pH meter (Corning model 12 or equivalent) equipped with combination electrode.

18. Measuring spoon, 1/4 teapoon volume.

3.2.5.5 Procedure (revised and updated from Smith et al., 1974)--

1. Add 1.250 g of less than 60 mesh rock or soil sample, 1/4 teaspoon decolorizing carbon, and 25 ml of $NaHCO_3$ solution to the 50 ml Erlenmeyer flask. Stopper the flask.

2. Shake for 30 minutes using a shaking speed of 2 on a Burrell wrist-action shaker.

3. Filter the suspension. NOTE: Shake flask before pouring suspension into filter funnel. If filtrate is yellow, add 1/4 teaspoon carbon, mix well and refilter. If filtrate is cloudy, filter using fine porosity filter paper.

4. Pipet 10 ml of filtrate into a 25 ml volumetric flask. Pipet 10 ml of H_2O into a separate 25 ml volumetric flask (blank). NOTE: If necessary to interrupt work, stop here, stopper and refrigerate.

5. Slowly add, with a pipet or calibrated dispenser, 5 ml of ammonium molybdate solution and mix immediately holding the top of the flask tightly closed. NOTE: Gases are generated during this mixing. The pH of the solution after adding molybdate should be between 3.0 and 4.0. With some alkaline soils it may be necessary to add more acid in order to assure the indicated pH for consistent color development. However, with minesoils studied, 5 ml of molybdate has been sufficient and has avoided excess acidity with extremely acid samples.

6. Wash down neck of flask with a small amount of water and dilute to about 22 ml.

7. Pipet 1 ml of the dilute $SnCl_2$ solution into the flask, dilute to volume (25 ml) with distilled water, and mix contents immediately.

8. After 10 minutes but less than 20 minutes after adding the dilute $SnCl_2$ to the flask and mixing, measure the adsorbance (A) of the blue solution, using the colorimeter or spectrophotometer at 660 nm. Read and understand instructions for operating the instrument correctly before using.

TABLE 2. STANDARDS FOR SODIUM BICARBONATE EXTRACTABLE PHOSPHORUS

P concentration (ppm P)	Volume of dilute (2 ppm) P Standard (ml)	Volume of H_2O (ml)	Volume of $NaHCO_3$ (ml)
Blank	0	13	5
0.08	1	12	5
0.16	2	11	5
0.24	3	10	5
0.32	4	9	5
0.40	5	8	5
0.48	6	7	5
0.56	7	6	5
0.64	8	5	5
0.72	9	4	5
0.80	10	3	5
0.88	11	2	5
0.96	12	1	5
1.04	13	0	5

9. Prepare standard curve of P concentration as follows:

a. Using 25 ml volumetric flasks, prepare phosphorus standards from Table 2.

b. Develop the color as in steps 5 and 7.

c. Make a standard curve by plotting absorbance (A) vs. P concentration (ppm) on linear graph paper.

d. Find ppm in sample extract by finding absorbance (A) of the extract on the standard curve and reading ppm of P directly from the curve.

3.2.5.6 Calculations--

1. ppm P in the rock or soil = ppm (read from the curve) X 50.

NOTE: The 50 is obtained from the following equation:

50 = (25 ml extracting solution/1.25 g sample) X (25 ml final volume/10 ml extract).

2. pp2m P in the soil = (ppm P in soil) X 2.

3.2.6 HCl-Extractable, HNO_3-Extractable and Non-Extractable Total Sulfur

3.2.6.1 Principle--

In doubtful cases, as stated in 3.2.4.1, this method should be used to rule out HCl-extractable and non-extractable forms of sulfur which are not considered to be acid formers. The HNO_3-extractable sulfur is determined by calculations. This form of sulfur will react with oxygen to produce acid.

3.2.6.2 Comments--

It is necessary to remove chlorides and nitrates by water leachings after the hydrochloric and nitric acid (respectively) extractions before running total sulfur.

Care should be taken that no sample is lost by run over, splashing or breaking through the filter paper during all leachings.

3.2.6.3 Chemicals--

1. Hydrochloric acid (HCl), 2 parts acid to 3 parts water: Mix 400 ml of concentrated HCl with 600 ml of distilled water.

2. Nitric acid (HNO_3), 1 part acid to 7 parts water: Mix 125 ml of concentrated HNO_3 with 875 ml of distilled water.

3. Silver Nitrate ($AgNO_3$), 10%: Dissolve 10.0 g of $AgNO_3$ in 90 ml of distilled water. Store in amber bottle away from light.

4. Nessler's Solution (Fisher Scientific Co. No. So-N-24 or equivalent).

3.2.6.4 Materials--

1. Leco Induction Furnace and Automatic Sulfur Titrator as in 3.2.4.4.

2. Funnels, 28 mm I.D. polyethylene.

3. Filter paper, 5.5 cm glass fiber.

4. Flasks, Erlenmeyer, 250 ml.

5. Beakers, 100 ml.

6. Syringe.

7. Balance, can be read to 0.001 g.

3.2.6.5 Procedure (Revised and updated from Smith et al., 1974)--

1. Take three 0.500 g subsamples of less than 60 mesh material.

2. Take one subsample and analyze for total sulfur (see 3.2.4).

3. Taking care not to sharply crease the glass fibers, fold filter paper to fit a polyethylene funnel.

4. Place second subsample in filter. NOTE: Make sure all material is placed in the filter.

5. Place subsample and filter onto funnel holder in sink or other suitable pan which can receive outflow from funnel.

6. Using a syringe, pipette, or other graduated dispenser, add 2:3 HCl to almost the top of the filter paper. Caution: During this step and all other leaching steps, be careful not to lose any sample by runover, splashing, or breaking through the filter paper.

7. Repeat step 6 until a total of 50 ml of acid has been added.

8. Place funnel holder, containing funnel and subsample, over a 100 ml beaker.

9. Leach subsample with 50 ml of distilled and deionized water. Discard leachate. NOTE: Stop here if procedure cannot be completed in one day. CAUTION: Samples must be kept moist.

10. Leach subsample with another 50 ml of distilled and deionized water.

11. Test leachate for chlorides by adding 3 drops of 10% $AgNO_3$ with a dropper. NOTE: The presence of chlorides will be detected by a white precipitate.

12. Discard leachate and repeat steps 10 and 11 until no precipitate forms.

13. Discard leachate.

14. Air dry subsample and filter overnight.

15. Carefully fold glass fiber filter around the sample and transfer to a ceramic crucible for total sulfur analysis (see 3.2.4).

16. Place third subsample in a 250 ml Erlenmeyer flask. NOTE: Make sure all of the subsample is placed in the flask.

17. Add 50 ml of HNO_3 (1:7).

18. Let stand overnight at room temperature.

19. Taking care not to sharply crease the glass fibers, fold a filter to fit a polyethylene funnel.

20. Place a funnel holder over a sink or other suitable pan which can receive outflow from funnel.

21. Carefully pour subsample and acid from the Erlenmeyer flask into the funnel. NOTE: Do not get material above top of filter paper.

22. Repeat step 21 using distilled and deionized water to wash all materials remaining in the Erlenmeyer flask into the funnel.

23. Place funnel holder containing funnel and subsample over a 100 ml beaker. NOTE: Stop here if procedure cannot be completed in one day. CAUTION: Sample must be kept moist.

24. Leach subsample with 50 ml of distilled and deionized water. Discard leachate.

25. Leach subsample with another 50 ml of distilled and deionized water.

26. Test leachate for presence of nitrates by adding 3 drops of Nessler's Solution with a dropper. NOTE: If nitrates are present, the leachate will turn yellow within 30 seconds as seen against a white background.

27. Discard leachate and repeat steps 25 and 26 until no nitrates are detected.

28. Discard leachate.

29. Air dry subsample and filter overnight.

30. Carefully fold glass fiber filter around the sample and transfer to a ceramic crucible for total sulfur analysis (see 3.2.4).

3.2.6.6 Calculations--

1. HCl-extractable sulfur (mostly sulfates) = (Total sulfur of untreated sample) minus (Total sulfur after HCl treatment).

2. HNO_3-extractable sulfur (mostly pyritic sulfur) = (Total sulfur after HCl treatment) minus (Total sulfur after HNO_3 treatment).

3. Non-extractable sulfur (mostly organic sulfur) = Total sulfur after HNO_3 treatment.

3.2.7 Lime Requirement By Ca $(OH)_2$ Titration

3.2.7.1 Principle--

When calcium hydroxide is added to the soil, it initially reacts with and neutralizes any acidity in solution. The calcium hydroxide further reacts with the acidity contained on the soil particles. A time period of four days is required for the reaction to go to equilibrium. Because 5 ml of 0.04 N calcium hydroxide is equivalent to 1 ton of pulverized limestone per 1000 tons of material, various amounts can be added to the sample making this treatment similar to liming the soil. After the 4 day incubation period, pH determinations are made. A titration curve is drawn comparing pH to the amount of pulverized limestone per 1000 tons of material. From this curve the amount of pulverized limestone per 1000 tons of material can be determined to bring the soil to a pH of 6.5 (Dunn, 1943).

3.2.7.2 Comments--

The calcium hydroxide must be protected from carbon dioxide in the air by using soda lime or ascarite in a guard tube. The method is time consuming due to a 4 day incubation period; however, it is a reliable and accurate method for determining the lime requirement.

3.2.7.3 Chemicals--

1. Calcium hydroxide ($Ca(OH)_2$), 0.04 N, saturated solution: Dissolve 1.5 g $Ca(OH)_2$ (use some excess) and dilute to 1 liter with carbon dioxide-free water (see 3.2.3.3. No. 1). Filter to remove calcium carbonate ($CaCO_3$) and protect filtrate from CO_2 in the air with soda lime or ascarite in a guard tube.

2. Standard buffer solutions, pH - 4.00 and pH - 7.00.

3. Chloroform ($CHCl_3$).

3.2.7.4 Materials--

1. Flasks, Erlenmeyer, 250 ml with rubber stoppers.

2. Balance, can be read to 0.1 g.

3. pH meter (Corning model 12 or equivalent) with combination electrode.

3.2.7.5 Procedure--

1. Place 10 g samples of less than 60 mesh air-dry soil in 7 flasks.

2. Add $Ca(OH)_2$ at the rates of 1/2, 1, 2, 3, 4, 5, 6 tons of pulverized limestone per 1000 tons of material using 5 ml of 0.04 N $Ca(OH)_2$ as the equivalent of 1 ton of pulverized limestone per 1000 tons of material.

3. Dilute to 100 ml with distilled water.

4. Add three drops of chloroform to prevent microbial activity.

5. Allow suspensions to stand in stoppered flasks for 4 days with thorough shaking twice a day.

6. After 4 day incubation period, calibrate pH meter using pH 4.00 and 7.00 standard buffer solutions (see 3.2.2) and determine suspension pH. NOTE: Gently swirl the suspension to insure good electrode-suspension contact.

3.2.7.6 Calculations--

1. Construct a titration curve by plotting pH on the horizontal axis and tons of pulverized limestone per 1000 tons of material on the vertical axis.

2. Plot points and construct a best-fit curve through the points.

3. Draw a line vertically from pH 6.5 to the curve and put an (X) on the curve.

4. Draw a line horizontally from the (X) to the vertical axis.

5. Determine tons of pulverized limestone per 1000 tons of material needed to bring the soil to pH 6.5.

3.2.8 Lime Requirement By the Five Minute Boiling Method

3.2.8.1 Principle--

Calcium hydroxide neutralizes the acidity in solution first and then reacts with and neutralizes the acidity contained on the soil particles. This reaction time is greatly reduced by boiling the sample and calcium hydroxide mixture for 5 minutes and allowing it to cool before a measurement is taken. The procedure is similar to liming the samples, since 5 ml of 0.04 N calcium hydroxide is equivalent to 1 ton of pulverized limestone per 1000 tons of material. A titration curve is drawn comparing pH to tons of pulverized limestone per 1000 tons of material. The amount of pulverized limestone needed to bring the soil to a pH of 6.5 can be read directly from the curve. (Abruna and Vicente, 1955).

3.2.8.2 Comments--

Because of the 5 minute boiling period, the time element is reduced from 4 days ($Ca(OH)_2$ method) to about 1 hour.

The calcium hydroxide must be protected from carbon dioxide in the air by using soda lime or ascarite in a guard tube.

3.2.8.3 Chemicals--

1. Calcium hydroxide ($Ca(OH)_2$), 0.04 N, saturated solution: Dissolve 1.5 g $Ca(OH)_2$ (use some excess) and dilute to 1 liter with carbon dioxide-

free water (See 3.2.3.3 No. 1). Filter off calcium carbonate ($CaCO_3$) and protect from CO_2 in the air with soda lime or ascarite in a guard tube.

2. Standard buffer solutions, pH = 4.00 and pH = 7.00

3.2.8.4 Materials--

1. Flasks, Erlenmeyer, 250 ml.
2. Hot plate.
3. Thermometer, 0 - 100°C.
4. Water Tray.
5. Balance, can be read to 0.1 g.
6. pH meter, (Corning model 12 or equivalent) with combination electrode.

3.2.8.5 Procedure--

1. Place 10 g samples of less than 60 mesh air-dry soil in 7 flasks.

2. Add $Ca(OH)_2$ at the rates of 1/2, 1, 2, 3, 4, 5, 6 tons of pulverized limestone per 1000 tons of material using 5 ml of 0.04 $\underline{N}$ $Ca(OH)_2$ as the equivalent of 1 ton of pulverized limestone per 1000 tons of material.

3. Dilute with 50 ml of distilled water.

4. Boil on a hot plate for 5 minutes. NOTE: Intermittent stirring of the samples may be necessary to avoid excessive foaming.

5. Cool in water tray to 25°C.

6. Calibrate pH meter using pH 4.00 and 7.00 buffer solutions.

7. Immediately after cooling, determine pH of soil + water + 0.04 $\underline{N}$ $Ca(OH)_2$ suspension using a glass electrode. NOTE: Gently swirl the beaker to insure good electrode-suspension contact.

8. Record pH.

3.2.8.6 Calculations--

See 3.2.7.6.

3.2.9 Lime Requirement by the Woodruff Buffer Method

3.2.9.1 Principle--

The solution used is calcium acetate buffered by p-nitrophenol. An excess of the buffered solution (at pH 7.0) is added to the sample and allowed to equilibrate for an hour. The pH of the solution is read and the lime requirement is based on the drop in pH of the buffered solution. By allowing the buffer solution to stand in contact with the sample, calcium ions from the solution saturate the exchange complex and hydrogen ions go into solution, thus lowering the pH (Woodruff, 1948).

3.2.9.2 Comments--

The method is quick, reliable, and adaptable to use on soils of different exchange capacities. The Woodruff buffer solution is strongly buffered and may not accurately detect the lime requirement for weakly acid samples. On strongly acid minesoil samples, the Woodruff buffer method correlated with the $Ca(OH)_2$ titration procedure of determining lime requirement (West Virginia University, 1971).

3.2.9.3 Chemicals--

1. p-Nitrophenol ($NO_2C_6H_4OH$).

2. Calcium acetate ($Ca(CH_3COO)_2 \cdot H_2O$).

3. Magnesium oxide (MgO), heavy, powder, laboratory grade (Fisher M-50 or equivalent).

4. Standard buffer solutions, pH = 4.00 and pH = 7.00.

5. Woodruff buffer, stock solution: In a 10 liter glass bottle mix 80.0 g of $NO_2C_6H_4OH$, 400.0 g $Ca(CH_3COO)_2 \cdot H_2O$, 6.2 g MgO, and 4 liters of distilled water. Make to 10 liters with distilled water. Put on reciprocating shaker at low speed overnight. Filter solution. Adjust to pH 7.00 with HCl or MgO.

6. Woodruff buffer, dilute solution: Mix 20 ml of Woodruff buffer stock solution with 10 ml of distilled water.

3.2.9.4 Materials--

1. Glass bottle, 10 liter.

2. Shaker, horizontal reciprocating type, 6.3 cm (2.5 in) stroke, 120 strokes per minute.

3. Paper cup or beaker

4. Automatic pipet, 5 ml.

5. Stirrer.

6. pH meter (Corning model 12 or equivalent) with combination electrode.

7. Balance, can be read to 0.01g.

3.2.9.5 Procedure--

1. Place 5.0 g of less than 60 mesh sample in a paper cup or beaker.

2. Add 5 ml of distilled water and mix with the soil. Let stand and mix occasionally for 1 hour.

3. Calibrate pH meter using pH buffer solutions of 4.00 and 7.00 (See 3.2.1).

4. Using pH meter, record pH of soil + water mixture by placing an electrode into the sample while shaking the cup. This insures good electrode contact with the mixture. NOTE: Soil + water mixture equalling or exceeding pH 6.5 have a lime requirement of zero tons of pulverized limestone per 1000 tons of material.

5. Add 5 ml of the Woodruff buffer stock solution.

6. Stir or shake for at least 30 minutes.

7. Using the dilute Woodruff buffer solution, adjust meter to a reading of exactly pH = 7.0.

8. Read and record pH of soil + water + Woodruff buffer stock solution mixture while shaking the cup to insure a good electrode contact. Record as buffered pH reading.

3.2.9.6 Calculations--

1. pH depression = (7.0) - (buffered pH reading).

2. Lime Requirement (L.R.) in tons pulverized limestone/1000 tons of material = 0.5 X (pH depression).

3.2.10 Lime Requirement by S.M.P. Buffer

3.2.10.1 Principle--

By measuring a change in pH of a buffer caused by the acids in a soil, Shoemaker, McLean, and Pratt (1962) determined the lime requirement of a soil. The lime requirement is read directly from a table based on pH of a soil after the S.M.P. buffer has been added.

3.2.10.2 Comments--

The S.M.P. buffer is very reliable for soils with a 2 ton per 1000 ton of material lime requirement. It adapts well for acid soils with a pH below 5.8 containing less than 10% organic matter and having appreciable quantities of soluble aluminum.

A sensitivity of 0.1 pH unit is needed for the interpretation of this method. A difference of 0.1 pH unit will result in a lime requirement difference of 0.5 to 0.9 tons of lime per 1000 tons of material for mineral soils.

Increased exposure time causes greater acidity thus causing a greater lime requirement. Increases in organic matter and/or clay content increases absorption of acidic cations. Buffer modifications may be necessary to prevent interference from hydroxy-iron and hydroxy-aluminum polymers. Air-dry soils may be stored several months in closed containers without affecting the SMP pH measurement.

3.2.10.3 Chemicals--

1. Standard buffer solutions, pH = 4.00 and pH = 7.00

2. SMP buffer solution : Dissolve 1.8 g p-nitrophenol ($NO_2C_6H_4OH$), 2.5 ml triethanolamine ($C_6H_{15}NO_3$), 3.0 g potassium chromate (K_2CrO_4), 2.0 g calcium acetate ($Ca(CO_2CH_3)_2$), and 53.1 g calcium chloride ($CaCl_2 \cdot 2H_2O$) with distilled water and dilute to 1 liter. Filter through a fiberglass sheet if suspended material is present. Connect an air inlet with a 2.54 X 30.5 cm (1 x 12 in) cylinder of drierite, a 2.54 X 30.5 cm cylinder of ascarite, and a 2.54 X 30.5 cm cylinder of drierite in series.

3.2.10.3 Materials--

1. Cup, 50 ml. glass, plastic, or waxed paper of similar size.

2. Pipet, 10 ml capacity.

3. Shaker, horizontal reciprocating type, 6.3 cm (2.5 in) stroke, 250 strokes per minute.

4. pH meter (Corning model 12 or equivalent) with combination electrode.

5. Balance, can be read to 0.1 g.

3.2.10.5 Procedure--

1. Weigh 5 g of less than 60 mesh sample into a 50 ml cup.

2. Add 5 ml of distilled water. Mix for 5 seconds.

3. Wait for 10 minutes and read the soil pH (see 3.2.2).

4. Add 10 ml SMP buffer solution to the cup for mineral soils with a pH of 6.5 or less.

5. Shake for 10 minutes on reciprocating shaker at 250 strokes per minute or stir.

6. Let stand for 30 minutes.

7. Read pH of the soil-buffer solution to the nearest 0.1 pH unit (see 3.2.2).

TABLE 3. SOIL-SMP BUFFER pH AND CORRESPONDING LIME REQUIREMENT (L.R.) TO BRING MATERIAL TO pH 6.5*

pH	L.R. (Tons/1000 Tons)**	pH	L.R. (Tons/1000 Tons)**
6.9	0.3	5.8	8.1
6.8	1.0	5.7	8.9
6.7	1.8	5.6	9.6
6.6	2.4	5.5	10.4
6.5	3.1	5.4	11.1
6.4	3.9	5.3	11.7
6.3	4.6	5.2	12.5
6.2	5.3	5.1	13.2
6.1	6.1	5.0	14.0
6.0	6.0	4.9	14.7
5.9	5.9	4.8	15.5

*Adapted from Shoemaker, McLean, and Pratt, 1962.

**Agricultural ground limestone TNP at least 90%.

3.2.10.6 Calculations--

Determine lime requirement from Table 3.

3.2.11 Total Sulfur Estimation By Peroxide Oxidation

3.2.11.1 Principles--

Pyritic minerals begin to change into two new products when exposed to the atmosphere. The change may proceed slowly over a long period of time before the final products (yellowboy and sulfuric acid) are formed. The end product, "yellowboy," actually may form only when the sulfate is partially or completely neutralized by a basic substance. The chemical equation for this complete change in pyrite follows:

Pyrite + Oxygen + Water equals Yellowboy + Sufluric Acid

$$4FeS_2 + 15O_2 + 14H_2O = 4Fe(OH)_3 + 8H_2SO_4$$

Hydrogen peroxide greatly reduced the time needed for pyrite to oxidize to sulfuric acid and yellowboy.

3.2.11.2 Comments--

Alkaline materials interfere with the efficiency of hydrogen peroxide in oxidizing pyrite; therefore, overburden rock and minesoil samples containing carbonates need to be leached with acid and water as prescribed in steps 2 through 5 of the procedure.

When samples contain readily oxidizable organic matter, step 7 in the procedure may have to be repeated until the reaction stops.

The hydrogen peroxide used in this method must be 30% hydrogen peroxide. It must not contain stabilizers.

An important thing to remember is that this procedure works with fresh overburden and not with complex mixtures of minesoil material.

3.2.11.3 Chemicals--

1. Silver nitrate ($AgNO_3$), 10%: Dissolve 10.0 g of $AgNO_3$ with distilled water and make to a volume of 100 ml. Store in brown bottle away from light.

2. Hydrochloric acid (HCl), 2 parts acid to 3 parts water: Mix 400 ml of concentrated HCl with 600 ml of distilled water.

3. Hydrogen peroxide (H_2O_2), 30% (Fisher certified No. H-325 or equivalent).

4. Sodium hydroxide (NaOH), 1.0 N: Dissolve 40.0 g of NaOH pellets in carbon dioxide-free water (see 3.2.3.3. No. 1) and make to a volume of 1 liter. Protect from CO_2 in air with ascarite tube.

5. Sodium hydroxide (NaOH), 0.1 N: Dilute 10 ml of 1.0 N NaOH to a volume of 1 liter with carbon dioxide-free water (see 3.2.3.3. No. 1). Standardize solution (see 3.2.3.3. No. 4). Protect from CO_2 in air with ascarite tube.

3.2.11.4 Materials--

1. Sample, ground to pass a 60 mesh sieve.

2. Funnels.

3. Hotplate. NOTE: Bunsen burner may be substituted.

4. Thermometer, °C.

5. Beakers, 300 ml tall form.

6. Graduated cylinder, 25 ml.

7. Glass fiber filter (Reeve Angel 934AH or equivalent).

8. Burets, 50 ml capacity.

9. Balance, can be read to 0.01 g.

10. pH Meter (Corning Model 12 or equivalent) with combination electrode.

3.2.11.5 Procedure (modified and updated from Smith et al., 1974)--

1. Weigh 2.00 grams of less than 60 mesh sample.

NOTE: If the sample contains no carbonates and no sulfates, and the paste pH is less than 5.5, then steps 2 through 5 can be eliminated and procedure can be continued at step 6.

2. Place sample into a funnel fitted with filter paper and leach with 200 ml of 2:3 HCl in funnel-full increments.

3. Leach sample with distilled water (in funnel-full increments) until effluent is free from chloride as detected by 10% silver nitrate. Note: Add three drops of silver nitrate. If a white precipitate forms, chlorides are present.

4. Air dry filter and sample overnight, or place in 50°C forced air oven until dry.

5. Carefully scrape dried sample from filter surface and mix sample.

6. Place sample in a 300 ml tall form beaker.

7. Add 24 ml of 30% H_2O_2 and heat beaker on hotplate until solution is approximately 40°C. Remove beaker from hotplate and allow reaction to go to completion as shown when bubbling ceases. NOTE: Three blanks for each batch of samples should be handled in the same manner. CAUTION: Initial reaction may be quite turbulent when samples contain more than 0.1% sulfur.

8. Add an additional 12 ml of H_2O_2 (30%) to beaker and allow reaction to go to completion as shown when bubbling ceases.

9. Place beaker on hotplate and heat to approximately 90 to 95°C, solution temperature, until any unreacted H_2O_2 left in beaker is destroyed as shown when bubbling ceases. Do not allow to go to dryness.

10. Wash down the sides of the beaker with distilled water and make the volume of the solution to approximately 100 ml.

11. Place beaker on the hotplate and heat the solution to boiling to drive off any dissolved CO_2, then cool the solution to room temperature.

12. Titrate the solution with 0.0100 N NaOH, that is free to CO_2 and protected from the atmosphere, to pH 7.0 using a pH meter.

3.2.11.6 Calculations--

1. meq H^+/100 g = (ml of NaOH) X (N of NaOH) X (100g/weight of sample).

2. % S = 0.0185 (meq H^+/100g) - 0.0806. (Grube, et al., 1973).

3. To convert percent sulfur (% S) to maximum $CaCO_3$ equivalents: Multiply %S by 31.25 to get tons $CaCO_3$ equivalent/1000 tons of material.

3.2.12 Double Acid Extractable Phosphorus, Potassium, Calcium, and Magnesium

3.2.12.1 Principle--

The method is a modified North Carolina Double Acid Method first published by Mehlich (1953) and then by Nelson, Mehlich and Winters (1953). Phosphorus, potassium, calcium, and magnesium are extracted from the sample using a solution containing dilute hydrochloric and sulfuric acid. Phosphorus concentration in the extract is determined using a colorimeter and calibration curve. The concentrations of potassium, calcium, and magnesium in the extract are determined using an atomic absorption spectrophotometer and calibration curve. The concentrations of each element can then be converted into pounds/1000 tons by calculations.

3.2.12.2 Comments--

With some soils a light to dark yellow color may develop in the extract. Decolorization is accomplished by the addition of activated charcoal in the extraction procedure. Lanthanum is added as a compensating element to remove phosphate and sulfate interference in the atomic absorption spectrophotometer methods for calcium and magnesium.

After the initial extraction, individual elements can be determined if data for all four elements are not required. Samples with elements higher in concentration than given in the calibration curves must be diluted and the resulting reading multiplied by the dilution factor.

3.2.12.3 Chemicals--

1. Hydrochloric acid (HCl), concentrated.

2. Sulfuric acid (H_2SO_4), concentrated.

3. Extracting solution: To make 0.05 N HCl and 0.025 N H_2SO_4, measure about 10 liters deionized water into an 18 liter pyrex bottle. Add 12 ml H_2SO_4 (96%) and 73 ml HCl (37%). Make to 18 liters with distilled water and mix thoroughly by shaking. Allow 12 hours to come to equilibrium.

4. Ammonium molybdate ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$).

5. Ammonium vanadate (NH_4VO_3).

6. Nitric acid (HNO_3), 1 N: Dilute 64 ml of concentrated HNO_3 (69.5%) to 1 liter with distilled water.

7. Molybdate - Vanadate solution: Dissolve 25 g of $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ in 500 ml of distilled water. Dissolve 1.25 g of NH_4VO_3 in 500 ml of 1 N HNO_3. Store in separate bottles. Mix equal volumes of these solutions (1 ml required per sample). Prepare fresh mixture each week.

8. Monobasic potassium phosphate (KH_2PO_4).

9. Phosphorus standard solution: Dissolve 0.1098 g of KH_2PO_4 in 500 ml of extracting solution. Dilute to 1 liter with extracting solution.

10. Potassium atomic absorption standard (1000 ppm).

11. Calcium atomic absorption standard (1000 ppm).

12. Magnesium atomic absorption standard (1000 ppm).

13. Potassium (K) standard stock solution (100 ppm): Place 10 ml of potassium atomic absorption standard (1000 ppm) in a 100 ml volumetric flask. Bring to volume with deionized water. Make fresh daily.

14. Calcium (Ca) standard stock solution (200 ppm): Place 20 ml of calcium atomic absorption standard (1000 ppm) in a 100 ml volumetric flask. Bring to a volume with deionized water. Make fresh daily.

15. Magnesium (Mg) standard stock solution (100 ppm): Place 10 ml of magnesium atomic absorption standard (1000 ppm) in a 100 ml volumetric flask and dilute to volume with deionized water. Make fresh daily.

16. Lanthanum chloride ($LaCl_3\cdot 6H_2O$), 5%: Dissolve 127 g of $LaCl_3\cdot 6H_2O$ with deionized water and bring to a volume of 1 liter.

17. Activated charcoal (Darco G-60 or equivalent).

3.2.12.4 Materials--

1. Atomic absorption spectrophotometer (Perkin-Elmer Model 403 or equivalent).

2. Colorimeter (Bausch and Lomb Spectronic 20 or equivalent).

3. Flasks, Erlenmeyer, 50 ml.

4. Flasks, volumetric, 100 ml.

5. Flasks, volumetric, 200 ml.

6. Pipet, 1 ml.

7. Pipet, 2 ml.

8. Shaker, horizontal reciprocating type, 6.35 cm (2.5 in) stroke with 120 strokes per minute.

9. Filter paper (Whatman 40 or equivalent).

10. Pyrex bottle, 18 liters.

11. Pyrex bottle, 8 liters.

12. Balance, can be read to 0.1 g.

3.2.12.5 Procedure--

1. Place 5.0 g of less than 60 mesh sample in a 50 ml Erlenmeyer flask. Add 0.2 g of activated charcoal. Prepare two blanks using only 0.2 g of activated charcoal.

2. Add 25 ml of extracting solution and shake for 5 minutes on the reciprocating shaker at 120 strokes per minute.

3. Filter using filter paper and save filtrate for P, K, Ca, and Mg determinations. NOTE: If filtrate is cloudy, refilter.

4. Subdivisions 3.2.12.5.1 through 3.2.12.5.3 include the determination of individual elements.

3.2.12.5.1 Phosphorus (P)--These steps are used for the determination of phosphorus.

1. Turn on colorimeter 15 minutes before use and adjust according to instruction manual.

2. Pipet 4 ml of filtered extract into a colorimeter tube.

3. Add 1 ml of molybdate-vanadate solution and allow to stand 10 minutes.

4. Mix by inverting tube and shaking by hand for a few seconds.

5. Place tube in instrument and read percent transmission (% T).

6. Using % T, determine the ppm available P from a calibration curve prepared as follows: (A) To separate colorimeter tubes, add the amounts of chemicals given in Table 4; (B) Treat as outlined in 3.2.12.5.1 steps 2-5; (C) Plot ppm on the horizontal axis and % T on the vertical axis. NOTE: If sample does not fall on calibration curve, samples must be diluted and results multiplied by the dilution factor. The dilution factor is obtained by taking the final volume and dividing it by the initial aliquot.

TABLE 4. PHOSPHORUS (P) STANDARDS

Phosphorus Standard Solution (ml)	Extracting Solution (ml)	Molybdate-Vanadate Solution (ml)	Phosphorus in Standard (ppm)
0.0	4.0	1.0	0.0
0.5	3.5	1.0	2.5
1.0	3.0	1.0	5.0
1.5	2.5	1.0	7.5
2.0	2.0	1.0	10.0
2.5	1.5	1.0	12.5
3.0	1.0	1.0	15.0
3.5	0.5	1.0	17.5
4.0	0.0	1.0	20.0

3.2.12.5.2 Potassium (K)--These steps are used for the determination of potassium.

1. Set the atomic absorption spectrophotometer unit on emission mode following the instrument's instruction manual.

2. Use the extractant for zero setting.

3. Put the extracted sample solution under the aspirating tube and record readings.

4. Determine ppm of K in the sample from the calibration curve prepared as follows: (A) Into separate 100 ml volumetric flasks, dilute the K standard stock solution with extracting solution for a range of 0 to 80 ppm increments; (B) Take reading with the atomic absorption spectrophotometer; (C) Plot available K (ppm) on the horizontal axis and instrument reading on the vertical axis; (D) Plot a curve through the points. NOTE: If samples do not fall on the calibration curve, dilute samples with extracting solution and multiply results by dilution factor. The dilution factor is obtained by dividing the final volume by the initial aliquot.

3.2.12.5.3 Calcium (Ca) and magnesium (Mg)--These steps are used for the determination of calcium and magnesium.

1. Adjust the atomic absorption spectrophotometer following the instrument instruction manual.

2. Pipet 1.0 ml of sample extract and blank into separate 100 ml volumetric flasks. Add 1.0 ml of 5% $LaCl_3 \cdot 6H_2O$ to each flask.

3. Bring to volume with extracting solution and mix by hand shaking.

4. In separate 100 ml volumetric flasks, prepare the calcium standards as shown in Table 5. Aspirate each standard into the instrument until a steady reading is obtained. Record reading.

5. Make a calibration curve plotting Ca (ppm) on the horizontal axis and instrument reading on the vertical axis. Plot a curve through the points.

6. Into separate 200 ml volumetric flasks, prepare the magnesium standards as shown in Table 6. Aspirate each standard into the instrument until a steady reading is obtained. Record reading.

7. Make a calibration curve plotting extractable Mg (ppm) on the horizontal axis and instrument reading on the vertical axis. Plot a curve through the points.

8. Aspirate sample extracts into the atomic absorption spectrophotometer and record readings.

9. Determine ppm of calcium and magnesium from calibration curves. If samples do not fall within the range of the calibration curve, dilute sample with extracting solution and add 5% $LaCl_3 \cdot 6H_2O$, but not to exceed 1% La in the final dilution. Multiply results by dilution factor. The dilution factor is obtained by taking the final volume and dividing it by the initial aliquot.

3.2.12.6 Calculations--

1. Dilution factor (DF) equals 1 unless the samples have to be diluted to fall within the range of the standard curve. The dilution factor is obtained by taking the final volume and dividing it by the initial aliquot.

2. ppm P in the soil = ppm (read from the curve) X 6.25 X DF. NOTE: The 6.25 is obtained from the following equation: 6.25 = 25 ml extracting solution/5 g sample) X (5 ml final volume/4 ml extract).

3. ppm K in the soil = ppm (read from the curve) X 5 X DF. NOTE: The 5 is obtained from the following equation: 5 = (25 ml extracting solution)/(5 g sample).

TABLE 5. CALCIUM (Ca) STANDARDS

Stock Ca Solution 200 ppm (ml)	$LaCl_3 \cdot 6H_2O$ (ml)	Extracting Solution (ml)	Calcium in Standard (ppm)
0.0	2.0	98.0	0.0
1.0	2.0	97.0	2.0
2.0	2.0	96.0	4.0
3.0	2.0	95.0	6.0
4.0	2.0	94.0	8.0
5.0	2.0	93.0	10.0

TABLE 6. MAGNESIUM (Mg) STANDARDS

Stock Mg Solution 100 ppm (ml)	$LaCl_3 \cdot 6H_2O$ (ml)	Extracting Solution (ml)	Magnesium in Standard (ppm)
0.0	4.0	196.0	0.00
0.5	4.0	195.5	0.25
1.0	4.0	195.0	0.50
1.5	4.0	194.5	0.75
2.0	4.0	194.0	1.00
2.5	4.0	193.5	1.25
3.0	4.0	193.0	1.50

4. ppm Ca in the soil = ppm (read from the curve) X 500 X DF. NOTE: The 500 is obtained from the following equation: 500 = (25 ml extracting solution/5 g sample) X (100 ml final volume/1 ml extract).

5. ppm Mg in the soil = ppm (read from the curve) X 500 X DF. NOTE: The 500 is obtained from the following equation: 500 (25 ml extracting solution/5 g sample) X (100 ml final volume/1 ml extract).

6. pp2m of element in the soil = (ppm of element in the soil) X 2.

3.2.13 Organic Carbon by Walkley-Black Method

3.2.13.1 Principle--

The method involves the oxidation of organic carbon by an oxidizing agent, potassium dichromate. The reaction is aided by the addition of sulfuric acid which generates heat. After the reaction is complete, the remaining dichromate is determined by titration with standard ferrous sulfate solution. From the amount of dichromate reduced, the amount of oxidized organic carbon can be calculated (Allison, 1965; Jackson, 1958).

3.2.13.2 Comments--

Some interference can result from chlorides, higher oxides of manganese and reduced iron. With the use of proper precautions, this interference can be eliminated or greatly reduced (Walkley, 1947; Jackson, 1958). Ferrous iron and chlorides tend to give positive or high organic carbon values, whereas, oxides of manganese tend to give negative or low values (Allison, 1965).

All samples should be ground in a porcelain or agate mortar. Iron or steel mortar is avoided because of the introduction of reducing material in the form of metallic iron.

3.2.13.3 Chemicals--

1. Potassium dichromate ($K_2Cr_2O_7$), 1 N: Dissolve 49.04 g $K_2Cr_2O_7$ (dried at 105°C) in distilled water and dilute to a volume of 1 liter.

2. Sulfuric acid (H_2SO_4), concentrated.

3. Ferroin solution, 0.025 M (available from Fisher Scientific Company)

4. Ferrous sulfate ($FeSO_4 \cdot 7H_2O$), 0.5 N: Dissolve 140.0 g of $FeSO_4 \cdot 7H_2O$ in distilled water. Add 15 ml of concentrated H_2SO_4 and allow to cool. Dilute to 1 liter with distilled water. Standardize reagent daily by titrating it against 10 ml of 1 N $K_2Cr_2O_7$.

3.2.13.4 Materials--

1. Porcelain or agate mortar and pestle.

2. Flasks, 500 ml, Erlenmeyer, wide-mouth.

3. Pipet, 10 ml.

4. Pipet, 20 ml.

5. Buret, 50 ml

6. Balance, can be read to 0.001 g.

7. Sieve, 0.25 mm (60 mesh) openings, nonferrous.

8. Buchner funnel.

9. Filter paper, (Whatman 40 or equivalent).

10. Weighing pans.

3.2.13.5 Procedure--

1. Grind air-dry samples to pass 60 mesh sieve with a porcelain or agate mortar.

2. Weigh and record tare weights of two clean and dry weighing pans.

3. In previously tared weighing pans, weigh 2.00 g (0.50 g of Horizon 1 and carbolith material) air-dry soil samples. NOTE: One sample is used for the procedure. The second sample is placed in an oven at 105°C for 16 hours, allowed to cool in a desiccator, and its oven-dry weight recorded. (See 3.2.13.6. No. 3).

4. Place weighted air-dry sample in a 500 ml Erlenmeyer flask.

5. Pipet exactly 10 ml of 1 $\underline{N}$ $K_2Cr_2O_7$ solution into the soil. Swirl flask gently until mixed.

6. Rapidly pipet 20 ml of concentrated H_2SO_4, directing the stream into the suspension. Mix by gentle rotation for 1 minute to insure complete contact of reagent with sample. NOTE: Avoid throwing soil up onto the sides of the flask and out of contact with the reagent.

7. Allow mixture to stand on an asbestos sheet for 30 minutes.

8. Dilute to 200 ml with distilled and deionized water.

9. Add 4 drops to 0.025 $\underline{M}$ Ferroin indicator.

10. Back titrate with 0.5 $\underline{N}$ ferrous sulfate solution from a buret. As the endpoint is approached, the solution has a greenish cast which changes to dark green. At this point, add ferrous sulfate drop by drop until the color changes sharply from blue to red (maroon color in reflected light against a white background). CAUTION: Discard and rerun with less soil

if 8 ml or more of the dichromate is reduced. If the endpoint cannot be clearly distinguished as described above, rerun sample and filter suspension using a buchner funnel before doing steps 9 and 10.

3.2.13.6 Calculations--

1. meq $K_2Cr_2O_7$ = (ml $K_2Cr_2O_7$ used) X ($\underline{N}$ $K_2Cr_2O_7$).

2. meq $FeSO_4$ = (ml $FeSO_4$ used) X ($\underline{N}$ $FeSO_4$).

3. Oven-dry weight of sample = (wt. oven-dry sample and tared pan) - (wt. of tared pan).

4. % organic carbon = [(meq $K_2Cr_2O_7$ - meq $FeSO_4$) X (0.003 X 100 X 1.33)]/ Oven dry sample wt.

3.2.14 Organic Carbon Determination By Low Temperature Ignition

3.2.14.1 Principle--

Water and hydroxides are driven off the sample by heating to 105°C. Organic matter is oxidized by heating at 400°C for 7 hours. The percent organic matter can be determined by weight loss.

3.2.14.2 Comments--

Mineral matter is assumed to be unchanged at the 400°C temperature range. For soils containing amorphous materials, the discrimination between organic and mineral matter is far from complete (Jackson, 1958).

3.2.14.3 Chemicals--

None required.

3.2.14.4 Materials--

1. Muffle furnace.

2. Drying oven.

3. Desiccator with drierite desiccant.

4. Balance, can be read to 0.01 g.

5. Crucibles or evaporating dishes.

3.2.14.5 Procedure (Modified from Jackson, 1958)--

1. Weigh a clean and dry crucible. Record tare weight (A).

2. Weigh 10.00 g of less than 60 mesh sample in tared crucible.

3. Place in oven and heat for 4 hours at 105°C.

4. Remove sample and allow to cool in desiccator.

5. Weigh sample. Record weight (B).

6. Place sample in oven and heat for 7 hours at 400°C.

7. Remove sample and allow to cool in desiccator.

8. Weigh sample. Record weight (C).

3.2.14.6 Calculations

1. Legend:

A = Tare weight of crucible.

B = Weight of sample and crucible after heating 4 hours at 105°C.

C = Weight of sample and crucible after heating 7 hours at 400°C.

D = Weight of sample after heating 4 hours at 105°C.

E = Weight of sample after heating 7 hours at 400°C.

2. D = B - A.

3. E = C - A.

4. Organic matter oxidized by heating = D - E.

5. % organic matter in sample = (Organic matter oxidized by heating/D) X 100.

3.2.15 Total Nitrogen by Kjeldahl Method

3.2.15.1 Principle--

In the Kjeldahl procedure, nitrogen is converted to ammonium ion by oxidation with concentrated sulfuric acid. With the addition of a catalyst such as copper, selenium, or mercury, this oxidation, which normally progresses very slowly, can be accelerated. Raising the boiling point by the addition of such salts as sodium sulfate or potassium sulfate also accelerates the reaction.

The ammonium ion produced by this oxidation is determined by making the solution strongly alkaline with sodium hydroxide, the liberated ammonia is distilled into a boric acid solution. The resulting ammonium borate is back titrated to boric acid with a standard acid (Bremner, 1965; Winkler, 1913).

3.2.15.2 Comments

Continuous boiling of the concentrated sulfuric acid and Kel-pak mixture for several hours requires insulation and venting of the system so the sulfuric acid condenses about one-third of the way up the digestion flask neck.

Materials adhering to the walls must be dislodged and brought into contact with the acid by rotation of the flask. Clay soils are particularly troublesome because clay promotes splattering. With the addition of glass beads, bumping during digestion can usually be eliminated. Optimum digestion temperature is between 360 and 400°C. Loss of nitrogen may occur if heated above 410°C.

3.2.15.3 Chemicals--

1. Kel-pak powder No. 3 ($HgO + K_2SO_4$) (available from Matheson Scientific Co.).

2. Sulfuric acid (H_2SO_4), concentrated.

3. Sulfuric acid (H_2SO_4), dilute (approximately 0.1 N): Dilute 44.8 ml of concentrate H_2SO_4 to 16 liters with distilled water.

4. Sodium hydroxide (NaOH), 45% with sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$): Under a fume hood in a rubber bucket mix 4545.9 g of NaOH flakes (for nitrogen determination) with 438.0 g of $Na_2S_2O_3 \cdot 5H_2O$. Dissolve and dilute to 11.355 liters (3 gal) with carbon dioxide-free water (See 3.2.2.2 No. 1). Cool overnight and siphon into dispensing apparatus. Protect from CO_2 in the air with soda lime or ascarite in a guard tube.

5. Boric acid (H_3BO_3), 4%: Dissolve 720.0 g of H_3BO_3 in distilled and deionized water on a hot plate. Dilute to 18 liters with distilled and deionized water. Add 60 ml of Bromocresol green-methyl red indicator (see below).

6. Bromocresol green-methyl red indicator: Mix 0.5 g of bromocresol green and 0.2 g methyl red with 100 ml of ethyl alcohol (90%). Adjust to medium color (brown) with a few drops of weak NaOH.

7. Zinc (Zn), granular.

3.2.15.4 Materials--

1. Kjeldahl electric digestion manifold

2. Kjeldahl electric distillation rack.

3. Room equipped with exhaust fan.

4. Flasks, Kjeldahl, 800 ml.

5. Flasks, Erlenmeyer, widemouth, 500 ml, marked at 230 ml.

6. Sieve, 20 mesh.

7. Balance, can be read to 0.1 g.

8. Asbestos gloves.

3.2.15.5 Procedure--

1. Place 10 g unground sample (sieved to 20 mesh) wrapped in filter paper in Kjeldahl flask. Also prepare two blanks without soil, but containing filter paper.

2. Add 2 packets of No. 3 Kel-pak.

3. Turn on exhaust fan.

4. Add 40 ml concentrated H_2SO_4. NOTE: While rotating flask, run acid down side to carry down sample.

5. Mix contents by gentle swirling and place flask carefully on Kjeldahl rack.

6. When all flasks are in place, set all knobs so that a moderate boiling and digestion of the sample can be seen.

7. After 30 minutes increase heat to a rapid boil for 30 minutes so that sulfur dioxide can be released and to insure complete digestion of the sample.

8. Rotate flasks 180° and continue heating until all the black organic matter is digested (usually about 1 hour).

9. Allow sample to cool on digestion rack and stopper. CAUTION. Do not place stopper in hot flask as it may implode upon cooling.

10. Let stand until solution reaches room temperature and cautiously add 300 ml distilled water to each flask. NOTE: Rotate flasks while pouring to wash neck.

11. Swirl flasks gently to dissolve crystals.

12. Add 1/4 teaspoon granular zinc to each flask.

13. Pour 30 ml H_3BO_3 (4% containing indicator) into 500 ml wide mouth Erlenmeyer flasks. NOTE: One required for each sample and blank and numbered to correspond to each Kjeldahl flask.

14. Place Erlenmeyer flasks on Kjeldahl distillation rack. NOTE: Top of glass delivery tube must be below surface of H_3BO_3.

15. Turn condenser water switch to manual. After 30 minutes turn water switch to automatic if unit is so equipped.

16. Add 133 ml NaOH (45%) slowly to each Kjeldahl flask. NOTE: Allow NaOH to run down side of flask so that it lies on the bottom.

17. Place each flask on Kjeldahl distillation rack as NaOH is added, using steps 18-21.

18. Wet hands with distilled water and apply water to rubber stoppers.

19. Place stopper securely in flask. Set flask on burner.

20. As soon as flask is in position, turn burner switch to make a moderate boil but not enough to cause solution to boil into flask neck.

21. Swirl flask to mix NaOH layer with the rest of the sample solution and set back in position making sure stopper is tight.

22. When 200 ml has distilled into receiving flask, set receiving flask (Erlenmeyer) down and turn off heat. CAUTION: Be sure to set flask down before turning off heat or distillate may suck back through condensers. NOTE: Distillate color should be green or dark blue.

23. Wash delivery tube with a small stream of distilled water from a wash bottle before removing receiving flask.

24. When cool, titrate distillate with 0.1 $\underline{N}$ H_2SO_4 until solution becomes clear and then turns pink.

25. Record reading.

3.2.15.6 Calculations--

1. Average of sample blanks = [reading (blank 1) + reading (blank 2)]/2.

2. Corrected sample reading = (sample reading) - (average of sample blanks).

3. Constant = ($\underline{N}$ acid) X (meq. wt. of N) X (100) X (1/wt. of sample); where $\underline{N}$ acid = 0.1, meq. wt. of nitrogen = 0.014, and 100 changes constant to percent.

The equation can then be written:

Constant = (0.1) X (0.014) X (100) X (1/wt. of sample), which can be simplified to:

Constant = (0.14) X (1/wt. of sample).

4. % nitrogen = (corrected sample reading) X constant.

3.2.16 Calcium Saturation Cation Exchange Capacity

3.2.16.1 Principle--

Cation exchange capacity (CEC) is defined as the sum of the exchangeable cations in a soil. Several methods are used for determining the CEC of a soil.

In this method, a solution of calcium chloride is used to saturate the soil exchange complex and remove all other exchangeable cations from the exchange sites. Calcium is then removed from the exchange complex by saturating the soil with magnesium acetate. By determining the amount of calcium in the magnesium acetate extract, the CEC of the soil can be measured.

3.2.16.2 Comments--

Soils with a pH greater than 5.5 or surface soils less than pH 5.5 which have been treated with lime must be pretreated with 1.0 N sodium acetate (pH 5.0) to move free carbonates (Jackson, 1958 pp. 62-63). To avoid this pretreatment, sodium saturated CEC (see 3.2.17) can be used.

Since calcium chloride is used to saturate the soil instead of a buffered acetate, the pH of the soil is not affected and the CEC is determined at the actual pH of the soil. This is important because it is well known that as pH rises the CEC increases (Coleman and Thomas, 1967).

3.2.16.3 Chemicals--

1. Calcium chloride ($CaCl_2 \cdot 2H_2O$), 1 N: Dissolve 147.03 g $CaCl_2 \cdot 2H_2O$ and dilute to 1 liter with distilled water.

2. Methanol (CH_3OH), 95%: Dilute 950 ml of methanol with 50 ml distilled water.

3. Magnesium acetate ($Mg(OAc)_2$), 1 N: Dissolve 107.25 g of $Mg(OAc)_2$ and dilute to 1 liter with distilled and deionized water.

4. Calcium atomic absorption standard (1000 ppm).

5. Calcium (Ca) standard stock solution (100 ppm): Pipet 10 ml of calcium atomic absorption standard (1000 ppm) in a 100 ml volumetric flask. Bring to volume with deionized water. Make fresh daily.

6. Silver nitrate ($AgNO_3$), 0.1%: Dissolve 0.10 g of $AgNO_3$ and dilute to 100 ml with distilled water. Store in brown bottle.

7. Lanthanum chloride ($LaCl_3 \cdot 6H_2O$), 5%: Dissolve 127 g of $LaCl_3 \cdot 6H_2O$ with deionized water and make to a volume of 1 liter.

3.2.16.4 Materials--

1. Balance, can be read to 0.0001 g.

2. Centrifuge tubes, 100 ml.

3. Rubber stoppers (to fit centrifuge tubes).

4. Shaker, horizontal reciprocating type, 6.35 cm (2.5 in) stroke, 120 strokes per minute.

5. Centrifuge (International Equipment Company Model K with No. 279 head or equivalent centrifuge and 12-place head).

6. Graduated cylinder, 100 ml.

7. Beaker, 100 ml.

8. Dropper bottle.

9. Bottle, polyethylene, 100 ml (one needed per sample).

10. Atomic Absorption unit (Perkin-Elmer Model 403 or equivalent).

11. Flasks, volumetric, 100 ml (7 required for standards).

12. Desiccator with drierite drying agent.

3.2.16.5 Procedure (Modified from Rich, 1961)--

1. Weigh 5 g of less than 60 mesh soil into a 100 ml centrifuge tube.

2. Add 50 ml of 1 N $CaCl_2$.

3. Stopper centrifuge tube and shake horizontally for 45 minutes on a reciprocating shaker insuring that the solid material in the bottom of the tube is completely dispersed.

4. Remove stopper and centrifuge suspension until clear (at least 5 minutes at 2000 RPM).

5. Pour off clear solution.

6. Repeat steps 2 through 5 two more times.

7. Add 50 ml of distilled water to the soil in the centrifuge tube.

8. Stopper and shake horizontally for 15 minutes on a reciprocating shaker insuring that the solid material in the bottom of the tube is completely dispersed.

9. Remove stopper and centrifuge for at least 5 minutes at 2000 RPM. Pour off clear solution.

10. Repeat steps 7 through 9 one more time.

11. Add 50 ml of 95% methanol to the soil in the centrifuge tube.

12. Stopper and shake horizontally for 15 minutes on a reciprocating shaker insuring that the solid material in the bottom of the tube is completely dispersed.

13. Remove stopper and centrifuge for at least 5 minutes at 2000 RPM.

14. Repeat steps 11 through 13 one more time.

15. Repeat steps 11 through 14, but pour clear solution into a 100 ml beaker.

16. Add a few drops of 0.1% $AgNO_3$ to the solution in the beaker. NOTE: If no precipitations occur, no further washing with methanol is required. If precipitation occurs, repeat steps 15 through 16 until no precipitation occurs.

17. Dry soil in the centrifuge tube in a drierite desiccator.

18. Weigh 0.5000 g dry, Ca-saturated soil into a 100 ml centrifuge tube.

19. Add 50 ml of 1 N $Mg(OAc)_2$.

20. Stopper and shake horizontally for 16 hours on a reciprocating shaker.

21. Remove stopper and centrifuge suspension until clear (for at least 5 minutes at 2000 RPM).

22. Pour solution into a 100 ml polyethylene bottle. Add 1.0 ml of 5% $LaCl_3 \cdot 6H_2O$ and cap bottle. NOTE: This solution will be used for Ca determination by atomic absorption.

23. Prepare CEC determination standards from Table 7.

24. Aspirate the standards on the atomic absorption unit following the instruction manual of instrument.

25. Make a standard curve plotting ppm of calcium on the horizontal axis and instrument reading on vertical axis.

26. Analyze samples for calcium and determine ppm of calcium from the prepared curve. NOTE: If unknown does not fall within the range of the standard curve, dilute sample with 1 N $Mg(OAc)_2$ and add 5% $LaCl_3 \cdot 6H_2O$, but not exceeding 1% La in the final dilution. The dilution factor is obtained by taking the final volume and dividing it by the initial aliquot.

3.2.16.6 Calculations--

1. Legend:

A = ppm of calcium as read from standard curve.

TABLE 7. STANDARDS FOR CALCIUM CEC DETERMINATION

Flask No. (100 ml)	ml of Ca stock solution (100 ppm)	ml of $Mg(OAc)_2$ (1 N)	ml of $LaCl_3 \cdot 6H_2O$ (5%)	Represented Ca (ppm)
1	0.0	98.0	2.0	0
2	1.0	97.0	2.0	1
3	2.0	96.0	2.0	2
4	4.0	94.0	2.0	4
5	6.0	92.0	2.0	6
6	8.0	90.0	2.0	8
7	10.0	88.0	2.0	10

DF = dilution factor, which is 1 if no dilution was necessary to read within the range of the standard curve. The dilution factor is obtained by taking the final volume and dividing it by the initial aliquot.

2. CEC (meq/100 g) = (A) X (DF) X (0.51), where the 0.51 is derived from the equation: (ppm/1,000,000) X (volume extracting solution/sample wt.) X (1000 meq per eq/eq. wt of Ca) X 100 g basis. NOTE: The volume of the extracting solution = 50 ml extracting solution + 1 ml $LaCl_3 \cdot 6H_2O$ = 51 ml.

3.2.17 Sodium Saturation Cation Exchange Capacity

3.2.17.1 Principle--

In this method, the soil is saturated with a solution of sodium acetate to replace all other exchangeable cations on the exchange sites with sodium. Sodium is then removed from the exchange complex by saturating the soil with an ammonium acetate solution. CEC is measured by determining the amount of sodium in the ammonium acetate extract.

3.2.17.2 Comments--

This method is used for both calcareous and noncalcareous soils. In minesoils, it is recommended that the sodium acetate method for determining CEC be used. Minesoils with a pH as low as 5.5 can contain free carbonates which interfere with the CEC determination by calcium saturation.

Cation exchange capacity may also be determined using ammonium acetate as a saturating solution; however, because of variable amounts of calcium carbonate and gypsum present in minesoils and their solubility in ammonium acetate, it is recommended that either sodium acetate or calcium chloride saturation be used for determining CEC. Solubility of calcium carbonate in 1 N sodium acetate at pH 8.2 is much lower than it is in neutral 1 N ammonium acetate.

Interferences occur in the sodium determination with some atomic absorption units. This interference can usually be corrected by the addition of 2,000 ppm of potassium to both the standards and the unknowns.

3.2.17.3 Chemicals--

1. Sodium acetate (NaOAc), 1.0 N: Dissolve 136 g of NaOAc in distilled water and dilute to 1 liter. NOTE: The pH of this solution should be 8.2. If needed, add a few drops of acetic acid or NaOH solution to adjust the pH to 8.2.

2. Ammonium acetate (NH_4OAc), 1.0 N: Dilute 114 ml of glacial acetic acid (99.5%) with distilled water to a volume of approximately 1 liter. Then carefully add 138 ml of concentrated ammonium hydroxide (NH_4OH) and slowly add distilled water to obtain a volume of approximately 1980 ml. Check the pH of the solution and add more NH_4OH as needed to obtain a pH of 7.0. Dilute the solution to a volume of 2 liters with distilled water.

3. Isopropyl alcohol, 99%.

4. Potassium stock solution, 10,000 ppm: Dissolve 19.07 g of potassium chloride (KCl) in 1 liter of deionized water.

5. Standard sodium solution, 1000 ppm, atomic absorption spectroscopy grade.

3.2.17.4 Materials--

1. Centrifuge tubes, 50 ml, round bottom polypropylene.

2. Rubber stoppers (to fit centrifuge tubes).

3. Shaker, horizontal reciprocating type, 6.35 cm (2.5 in.) stroke, 120 strokes per minute.

4. Centrifuge (International Equipment Company Model K with No. 279 head or equivalent centrifuge and 12-place head).

5. Volumetric flasks, 100 ml.

6. Atomic absorption spectrophotometer (Perkin-Elmer model 403 or equivalent).

7. Balance, can be read to 0.01 g.

3.2.17.5 Procedure--

1. Weigh 4.0 g of less than 60 mesh material and transfer to 50 ml centrifuge tube. NOTE: If the material is very coarse textured (loamy sand or sand), a 6.0 g sample is used.

2. Record weight of sample (A).

3. Add 33 ml of 1.0 N NaOAc solution to the centrifuge tube.

4. Stopper the tube and shake in a reciprocating shaker at 120 strokes per minute for 5 minutes insuring that the solid material in the bottom of the tube is completely dispersed.

5. Unstopper the tube and centrifuge until the supernatant liquid is clear (at least 5 minutes at 2000 RPM). Decant and discard the liquid.

6. Repeat steps 3 through 5 three more times.

7. Add 33 ml of 99% isoproply alcohol to centrifuge tube.

8. Stopper tube and shake on reciprocating shaker for 5 minutes insuring that the solid material in the bottom of the tube is completely dispersed.

9. Unstopper centrifuge tube and centrifuge it until the supernatant liquid is clear (at least 5 minutes at 2000 RPM). Then decant and discard the liquid.

10. Repeat steps 7 through 9 two more times.

11. Add 33 ml of 1 N NH_4OAc to centrifuge tube, stopper tube and shake for 5 minutes insuring that the solid material in the bottom of the tube is completely dispersed.

12. Unstopper tube and centrifuge until supernatant liquid is clear (at least 5 minutes at 2000 RPM).

13. Decant liquid into a 100 ml volumetric flask.

14. Repeat steps 11 through 13 two more times.

15. Fill the volumetric flask to the 100 ml mark using the 1N NH_4OAc solution.

16. Take 10 clean 100 ml volumetric falsks and label them 0, 5, 10, 20, 30, 40, 50, 60, 70, and 80 ppm sodium.

17. Pipet 0.5 ml of the 100 ppm sodium standard into the flask labeled 5 ppm sodium. Into the flasks labeled 10 through 80 ppm, pipet 1 ml through 8 ml, respectively, of the 1000 ppm sodium standard solution.

18. Dilute all flasks to volume with 1 N NH_4OA solution. NOTE: The flask labeled 0 ppm will contain only the 1 N NH_4OAc extracting solution.

19. Turn on the atomic absorption unit and wet it for emission mode. Read instruction manual carefully and set all operating parameters according to the instrument instruction manual.

20. After the atomic absorption unit is ready, zero the instrument using the 1 N ammonium acetate extracting solution, not distilled water. Aspirate standards and record readings.

21. Plot a standard curve using ppm sodium on the horizontal axis and the instrument readings on the vertical axis.

22. Record the instrument readings for all unknowns and read the concentration (B) of sodium from the standard curve. NCTE: If the unknown does not fall within the range of the standard curve which you have plotted, dilute the unknown with NH_4OAc and potassium stock solution using 2 ml of the potassium stock solution for every 10 ml of NH_4OAc. Then measure the amount of sodium present.

3.2.17.6 Calculations--

1. Legend:

A = Sample weight.

B = ppm of sodium as read from the standard curve.

DF = dilution factor, which is 1 or unity if no dilution of the unknown had to be made to get it to read within the range of the standard curve.

2. CEC (meq/100g) =

(B/1,000,000) X (DF) X (Vol. extracting solution/sample wt.) X (1000 meq/eq. wt Na) X 100g,

Where:

Vol. extracting solution = 100 ml

eq. wt of Na = 23.

The above equation can be reduced to:

CEC (meq/100g) = (B X DF X 10) / (23 X A).

3.2.18 Electrical Conductance of Soil Extract

3.2.18.1 Principle--

Pure water (water which contains no dissolved substances) is not a good conductor of an electrical current. Water becomes a better electric current conductor with the addition of dissolved salts. The amount of electric current conducted through this water is approximately proportional to the amount of salts dissolved in the water. Based on this fact, a measurement of the amount of electric current that is conducted by a soil extract will provide information as to the amount of salts present in the soil. This simple measurement provides an accurate indication of the concentration of ionized constitutents in the soil extract. The electrical conductivity of a soil extract is closely related to the sum of cations (or anions) as determined chemically. This measurement usually correlates closely with the total dissolved solids.

3.2.18.2 Comments--

Extracts to be used for electrical conductivity measurements should be taken from a saturated soil paste. Measuring the salt concentration of an extract obtained at the field moisture state would be an ideal method; however, it is much easier to obtain a soil extract from a saturated paste. This is extremely important when doing electrical conductivity measurements on a routine basis.

When making a saturated soil paste, some practice is necessary to obtain consistent results. Dried peat or muck usually require an overnight wetting period to obtain a satisfactory saturated paste. Add water to fine textured soils without stirring and allow the sample to wet slowly. This will enable the fine textured material to reach saturation without puddling occurring. Care must be taken not to overwet coarse textured soils. If water stands on the surface, the soil has been over saturated and a small additional amount of soil must be added.

The soil material used for electrical conductivity measurements should not be oven dried. Material should be air dried and ground to pass a 60 mesh sieve (see 3.1.2).

3.2.18.3 Chemicals--

1. Distilled water.

2. Potassium chloride (KCl), 0.01 N: Dissolve 0.7456 g of KCl in distilled water, and dilute with distilled water to 1 liter. This is the standard reference solution and at 25°C it has an electrical conductivity to 0.00141 mho/cm.

3. Sodium metaphosphate ($(NaPO_3)_6$), 0.1%: Dissolve 0.1 g of $(NaPO_3)_6$ (Fisher Scientific #S-333) in distilled water and dilute to 100 ml.

3.2.18.4 Materials--

1. Wheatstone bridge, alternating-current type, suitable for conductivity measurements. (Industrial Instruments Incorporated Model RC-16B2 or equivalent).

2. Conductivity cell, pipette-type, with platinized platinum electrodes. The cell constant should be approximately 1.0 reciprocal centimeter.

3. Flask, volumetric, 1000 ml.

4. Balance, can be read to 0.01 g.

5. Aluminum can with lid (large enough to contain sample).

6. Spatula.

7. Aluminum weighing pan.

8. Drying oven.

9. Dessicator, with silica gel dessicant.

10. Buchner type filtering funnel, 11 cm inside diameter.

11. Filter flask.

12. Filter paper (Whatman 42 or equivalent).

13. Vacuum source.

14. Graduated cylinder, 100 ml volume.

15. Pipette, measuring, 10 ml capacity.

3.2.18.5 Procedure (modified from U.S. Salinity Laboratory Staff, 1954)--

1. Weigh 400 g of air-dried soil. Transfer the soil to an aluminum can (with lid).

2. Add water to the sample in small increments by pouring the water down the side of the can. Water is added to the sample in this fashion until the saturation point of the soil is almost reached.

NOTE: Do not stir soil sample while adding water. Since water movement through puddled soil is very slow, the soil is allowed to wet by capillarity and then mixed to ensure against puddling.

3. Stir the wetted soil with a spatula until a condition of saturation is reached. Small amounts of water may be added while mixing to insure that the saturation point has been reached. NOTE: At saturation the soil paste glistens as it reflects light and the mixture slides off of the spatula easily.

4. After the mixing has been completed, place the lid on the aluminum can and let sample stand for 1 hour or more.

5. After sample has set for the required amount of time, check sample for saturation. NOTE: If the paste has stiffened or lost its glisten, add more water and mix it again. On the other hand, if free water has collected on the surface of the paste, add additional air-dry soil to absorb free water and remix the sample.

6. After a saturated paste has been obtained, remove a teaspoon-full of the saturated paste for oven-drying and replace lid. Allow the saturated soil paste to stand at least 4 hours.

7. Weigh an oven-dry aluminum weighing pan to the nearest 0.01 g. Record weight (A).

8. Place subsample of the saturated soil paste (from step 6) in aluminum weighing pan. Weigh pan and sample to the nearest 0.01 g. Record weight (B).

9. Place weighing pan and sample in an oven at 105°C for 16 hours (or overnight). Remove from oven and cool in a dessicator.

10. Weigh oven-dry sample and pan. Record weight (C).

11. After the saturated soil paste has stood for at least 4 hours (from step 6), transfer it to a Buchner funnel fitted with one sheet of Whatman #42 (or equivalent) filter paper.

12. Attach filter flask to vacuum source, apply vacuum, and collect filtrate. Terminate filtration when air begins to pass through the filter. NOTE: Refilter if filtrate is turbid.

13. Add one drop of 0.1% sodium hexametaphosphate solution for each 25 ml of extract.

14. Allow the standard 0.01 N KCl solution and the sample of the soil-water extract to adjust to room temperature. NOTE: As long as the temperature of the room is within the range of 20-30°C, the absolute temperature of the solutions are not important. However, it is extremely important that the standard solution and the extract be at the same temperature. If greater precision is required bring the standard solution and soil-water extracts to a temperature of 25°C in a constant temperature bath.

15. Turn on Wheatstone bridge and allow instrument to warm up.

16. When instrument is ready, rinse and fill the conductivity cell with the standard 0.01 N KCl solution.

17. Balance the wheatstone bridge according to the instruction manual provided by the manufacturer. Record the cell resistance (D) in ohms.

18. Rinse and fill the cell with the soil-water extract. NOTE: If the volume of the extract is limited, rinse the cell with distilled water followed by acetone. Dry the cell by drawing air through it until the acetone has evaporated. Allow the cell to come to room temperature.

19. Balance the bridge and record the cell resistance (E) in ohms.

3.2.18.6 Calculations--

1. Legend:

A = Weight of oven-dry weighing pan.

B = Weight of saturated soil and weighing pan.

C = Weight of oven-dry soil and weighing pan.

D = Initial cell resistance.

F = Final cell resistance.

2. % Moisture of sample at saturation = [(B-C)/(C-A)] X 100.

3. Electrical conductivity (EC) mmhos/cm, at 25°C = [(0.0014118 X D)/F].

4. Total cation concentration, meq/liter = 10 X (EC).

3.2.19 Sodium-Absorption-Ratio

3.2.19.1 Principle--

Plants growing in saline soils are affected by the salt concentrated in the soil solution. The principle cations present are calcium, magnesium, and sodium with small amounts of potassium. If the proportion of sodium is high, the alkali hazard is high. By making a soil-water extract and measuring the salt concentration of the extract, the salinity hazard of the soil can be determined.

3.2.19.2 Comments--

Lanthanum chloride must be added to both the standards and the extract to eliminate interferences in determining calcium and magnesium by atomic absorption. Interferences may also occur in the sodium determination and should be corrected by the addition of an excess (1000-2000 ppm) of potassium or lithium to both the standards and samples (see manuals supplied with atomic absorption unit).

3.2.19.3 Chemicals--

1. Calcium atomic absorption standard (1000 ppm).

2. Magnesium atomic absorption standard (1000 ppm).

3. Sodium atomic absorption standard (1000 ppm).

4. Lanthanum chloride ($LaCl_3 \cdot 6H_2O$), 5%: Dissolve 127 g of $LaCl_3 \cdot 6H_2O$ with deionized water and bring to a volume of 1 liter.

5. Sodium metaphosphate ($(NaPO_3)_6$), 0.1%: Dissolve 0.1 g of $(NaPO_3)_6$ (Fisher Scientific No. S-333) in distilled water and dilute to 100 ml.

3.2.19.4 Materials--

1. Atomic absorption spectrophotometer (Perkin-Elmer Model 403 or equivalent).

2. Flasks, volumetric, 100 ml.

3. Pipet, 1 ml.

4. Balance, can be read to 0.01 g.

5. Aluminum can with lid (large anough to contain sample).

6. Spatula.

7. Weighing pan.

8. Drying oven.

9. Desiccator.

10. Buchner filter funnel.

11. Filter paper (Whatman 42 or equivalent)

12. Vacuum source pulling a constant vacuum.

13. Bottle (to collect filtrate).

3.2.19.5 Procedure (modified from Bower and Wilcox, 1965; U.S. Salinity Laboratory Staff, 1954)--

1. Weight 400 g of air-dry soil. Transfer soil to an aluminum can (with lid).

2. Add water to the sample in small increments by pouring the water down the side of the can. Water is added to the sample in this fashion until the saturation point of the soil is almost reached. NOTE: Do not stir sample while adding water. Since water movement through puddled soil is very slow, the soil is allowed to wet by capillarity and then mixed to insure against puddling.

3. Stir the wetted soil with a spatula until a condition of saturation is reached. Small amounts of water may be added while mixing to insure that the saturation point has been reached. NOTE: At saturation the soil paste glistens as it reflects light and the mixture slides off of the spatula easily.

4. After the mixing has been completed, place the lid on the aluminum can and let stand for at least 1 hour.

5. After sample has set for the required amount of time, check sample for saturation. NOTE: If the paste has stiffened or lost its glisten, add more water and remix. If free water has collected on the surface, add additional air-dry soil to absorb the free water and remix.

6. After a saturation paste has been obtained, remove a teaspoonful of the saturated paste for oven-drying and replace lid. Allow the saturated soil paste to stand at least 4 hours.

7. Weigh an oven-dry aluminum weighing pan to the nearest 0.01 g. Record weight (A).

8. Place subsample of saturated soil paste (from step 6) in aluminum weighing pan. Weigh pan and sample to the nearest 0.01 g. Record weight (B).

9. Place weighing pan and sample in an oven at 105°C for 16 hours. Remove from oven and cool in dessicator.

10. Weigh oven-dry sample and pan. Record weight (C).

11. After the saturated soil paste has stood for at least 4 hours (from step 6), transfer it to a Buchner funnel fitted with one sheet of Whatman No. 42 (or equivalent) filter paper.

12. Attach filter flask to vacuum source, apply vacuum, and collect filtrate. Terminate filtration when air begins to pass through the filter. NOTE: Refilter if filtrate is turbid.

13. Add one drop of 0.1% sodium hexametaphosphate solution for each 25 ml of extract.

14. Take 10 clean 100 ml volumetric flasks and label them 0, 5, 10, 20, 30, 40, 50, 60, 70, and 80 ppm sodium.

15. Pipet 0.5 ml of 1000 ppm sodium standard into the flask labeled 5 ppm sodium. Into the flasks labeled 10 through 80 ppm, pipet 1 through 8 ml, respectively, of the 1000 ppm sodium standard solution.

16. Dilute all flasks to volume with deionized water. NOTE: The flasked labeled 0 ppm will contain only deionized water.

17. Turn on the atomic absorption unit and set it for emission mode. Read instrument instructions manual carefully and do all settings accordingly.

18. After the atomic absorption unit is ready, zero the instrument using the 0 ppm standard. Record the reading for each of the other standards.

19. Plot standard curve using ppm sodium on the horizontal axis and instrument reading on the vertical axis.

20. Measure the amount of sodium present in the unknowns. NOTE: If the unknown does not fall within the range of the standard curve, dilute with deionized water and remeasure the amount of sodium present. Record the dilution factor (DF). The dilution factor is obtained by taking the final volume and dividing it by the initial aliquot.

21. For each 100 ml of extract, or part thereof, of the volume found in step 13, add 2 ml of 5% $LaCl_3 \cdot 6H_2O$.

22. Prepare calcium and magnesium standards as shown in Table 8 using 100 ml volumetric flasks.

23. Set atomic absorption unit to absorption setting according to the instruments instruction manual.

24. After the atomic absorption unit is ready, zero the instrument using the 0 ppm standard (flask no. 1). Record the reading for each of the other standards.

25. Plot standard curves using ppm of element on the horizontal axis and instrument reading on the vertical axis.

26. Measure the amount of calcium and magnesium present in the unknowns. NOTE: If the unknown does not fall within the range of the standard curve, dilute with deionized water and add 5% $LaCl_3 \cdot 6H_2O$, but not to exceed 1% La in the final dilution. Remeasure the amount of calcium and magnesium present and record the dilution factor. The dilution factor is obtained by taking the final volume and dividing it by the initial aliquot.

3.2.19.6 Calculations--

1. Legend:

A = Weight of oven-dry weighing pan.

B = Weight of saturated soil and paste and weighing pan.

C = Weight of oven-dry soil paste and weighing pan.

2. Meq/l of Na = ppm of Na (read from curve)/23.00, where 23.00 is the equivalent weight of sodium.

3. Meq/l of Ca = ppm of Ca (read from curve)/20.04, where 20.04 is the equivalent weight of calcium.

4. Meq/l of Mg = ppm of Mg (read from curve)/12.16, where 12.16 is the equivalent weight of magnesium.

TABLE 8. CALCIUM AND MAGNESIUM STANDARDS FOR SODIUM-ADSORPTION RATIO

Flask No.	Calcium stock solution (100 ppm) (ml)	Magnesium stock solution (10 ppm) (ml)	$LaCl_3 \cdot 6H_2O$ (5%) (ml)	Deionized water (ml)	Represents ppm Ca	Represents ppm Mg
1	0.0	0.0	2.0	98.0	0.0	0.0
2	1.0	2.0	2.0	95.0	1.0	0.2
3	2.0	4.0	2.0	92.0	2.0	0.4
4	4.0	6.0	2.0	88.0	4.0	0.6
5	6.0	8.0	2.0	84.0	6.0	0.8
6	8.0	10.0	2.0	80.0	8.0	1.0
7	10.0	15.0	2.0	73.0	10.0	1.5

5. Sodium-adsorption-ratio = $Na^+/\sqrt{(Ca^{++} + Mg^{++})/2}$, where Na^+, Ca^{++}, and Mg^{++} refer to the concentrations of designated cations expressed in millequivalents per liter as found in calculations no. 2 through 4.

6. Saturated water percentage = [(B-C)/(C-A)] X 100.

3.3 MINERALOGICAL METHODS

3.3.1 Summary

Minerals occurring in overburden materials can be identified using a petrographic microscope or x-ray diffraction unit. Individual soil or rock grains are identified by placing the grains in an oil with a known index of refraction and examining them with the aid of a petrographic microscope. Individual grains and their relationships to surrounding grains are identified and examined in thin section using the petrographic microscope. Using x-ray diffraction, the types of clay minerals present in a sample can be determined.

All of the procedures require some technical knowledge for the mineral identification. A person experienced in the use of a petrographic microscope and/or x-ray diffraction instrument should make the identifications.

3.3.2 Identification of Grains by Immersion Method

3.3.2.1 Principle--

Many minerals may be identified by measuring their indices of refraction and then referring to determinative tables. The index may be measured by using the immersion method. Liquids of known index of refraction ranging from about 1.43 to 1.71 in steps of 0.01 should be available. The mineral grains to be identified are placed on a glass slide, covered with liquid of known index, and a small cover glass placed on top of the liquid. The grains are then observed under a petrographic microscope using a medium power objective. If the mineral grains have the same index as the liquid, they will be practically invisible. If the grains do not "match" the liquid, one can determine whether the grains have a higher or lower index than the liquid by the Becke Line Test. When a mineral grain is slightly out of focus, a narrow line of light known as the Becke line forms near the edge of the grain. The line is usually more conspicuous if light is reduced by partially closing the diaphragm in the substage. If the tube of the microscope is raised (or microscope stage lowered), the Becke line will move into the medium of higher index. In this way, it is possible to determine whether the grain has an index higher or lower than the liquid. As an example, if the grain is lower than the liquid, a new immersion is prepared using a liquid of lower index of refraction. If the grain still does not have the same index as the liquid, different liquids are used until a match is attained. In using a white light source, two Becke lines form when the grain and liquid are nearly matched--one line is yellowish and the other line bluish. When the microscope tube is raised, the brighter of these two lines moves toward the medium of higher index. The grain and liquid have the same index of refraction when the intensities of these two lines are the same.

3.3.2.2 Comments--

Amorphous material (no crystal structure) and isometric (cubic) crystals are said to be optically isotropic, having only one index of refraction which can be measured at any position of the microscope stage. All other minerals have two or more indices of refraction and are said to be anisotropic. Isotropic substances remain dark as the microscope stage is rotated with crossed nicols (upper polarizing element inserted). Anisotropic minerals, on the other hand, will generally be illuminated under crossed nicols, becoming dark every 90 degree turn of the microscope stage. These dark settings are called extinction positions. At these extinction positions the indices of refraction of anistropic minerals are measured.

Hexagonal and tetragonal minerals have two indices of refraction called n_O and n_E. A mineral is said to be positive when n_O is less than n_E and negative when n_O is larger than n_E. The index n_O can be measured on any grain by turning the stage to the low index extinction position in a positive mineral or to the high index position for a negative mineral. At the other extinction position an index called n_E' lying between n_O and the true n_E is obtained.

In positive minerals the highest value of n_E', as determined on several grains of the same mineral, is closest to true n_E. In a negative mineral, the lowest value obtained would be closest to n_E. Precise measurements of n_E require use of interference figures which are explained in standard optical mineralogy textbooks.

Orthorhombic, monoclinic, and triclinic minerals have three indices of refraction with the lowest index called n_X, the intermediate index n_Y, and the highest index n_Z. Exact determination of these indices involves somewhat complicated techniques which are explained in standard optical mineralogy textbooks. However, if several grains of the same mineral are examined, the lowest index obtainable on any of these grains will be fairly close to the n_X index. The highest index obtainable on the grains will be close to the n_Z index. The difference between the highest and lowest index in a given mineral is called birefringence.

Most minerals under crossed nicols will show spectral colors called interference colors. Interference colors result from the double refraction of light in the crystal. As the two rays emerge from the grain, they undergo interference as they combine in passing through the upper nicol. The color sequence is the same as in Newton Colors. The actual color observed depends on the thickness of the grain, its orientation, and difference between its highest and lowest index (birefringence). A mineral in randomly oriented grains of the same thickness will show all of the colors up to a certain maximum on the Newton Scale. This maximum color is very useful in identification. Although the general properties of two minerals may be quite similar, their interference colors may be clearly different.

Most of the 50-100 mesh constituents in a soil can be readily identified with the petrographic microscope by the immersion method using liquid 1.544. The most common minerals and rock particles with their distinguishing features are as follows:

1. Quartz can be distinguished from most other minerals by the fact that its low index (n_O) is always 1.544. The birefringence of quartz is weak and is similar to that of feldspar. However, unlike feldspar, quartz has no cleavage and is free of alteration or weathering to argillaceous or clayey material.

2. Chert, which is an aggregate of fine-grained quartz, may occur as grains in soil. Chert has the same optical properties as coarse quartz but under crossed nicols shows a mosaic or salt and pepper effect because of the diverse orientation of constituent quartz domains. Chert is distinguished from aggregates of clay which sometimes have a mosaic appearance by its lower index of refraction.

3. Orthoclase can be distinguished from most other minerals because its indices of refraction are noticeably lower than 1.544. The grain edges are commonly straight and parallel because of cleavage. Orthoclase is usually not as clear as quartz due to alteration. The birefringence of orthoclase is weak.

4. Microcline is similar to orthoclase but under crossed nicols shows spindle-shaped twin plates. Plates meet at right angles forming a grid-iron-like pattern.

5. Plagioclase commonly shows parallel bands or stripes under crossed nicols because of twinning. The indices of refraction of plagioclase vary with its composition. The more sodium-rich plagioclases have indices below 1.544 but not as low as potassium-rich feldspars. Plagioclases with a small amount of calcium have indices close to 1.544. The calcium-rich plagioclases have indices well above 1.544.

6. Muscovite occurs in colorless flakes with indices of refraction considerably higher than that of quartz or feldspar. In immersions, these flakes have a gray color under crossed nicols.

7. Biotite also occurs in flakes and in immersions under plain light is dark brown or less commonly green. Under crossed nicols hardly any light passes through the flakes.

8. Carbonate in the form of calcite or dolomite has a very high index in one extinction position and a low index near or below 1.544 in the other extinction position. This change in index as the stage is rotated is ordinarily very conspicuous and distinguishes carbonate from most other minerals. Under crossed nicols, carbonates have a unique pinkish tan color. Small carbonate particles mixed with clay may be recognized by introducing the substage condensing lens and crossing the nicols. Under these conditions, the carbonate will normally appear as bright specks.

9. Pyrite is opaque even with strong transmitted light obtained with the substage condensing lens. In reflected light pyrite has a brass yellow color and the crystal faces or polished surfaces look like metallic mirrors.

10. Limonite (goethite) is yellow or brown on thin edges under strong transmitted light and opaque in thicker masses. Under reflected light, limonite is yellowish brown to brown.

11. Hematite is opaque and black in reflected light where massive but commonly translucent and red in reflected light at thin edges of the material.

12. Sandstone fragments are recognized by the constituent grains of quartz which have the characteristic low index of 1.544 and gray to white interference colors. The grains may be bound together by cement of quartz, carbonate, clay or iron oxide.

13. Shale and mudstone usually show a fine layering due to parallel alignment of flakes of clay minerals. The index of refraction is moderately high (distinctly higher than 1.544) and interference colors are white to yellowish white. The fragments go to extinction when the layering is parallel to the polarizing elements because of the parallel alignment of the constituent clay minerals.

14. Limestone fragments have a high index of refraction which causes the fragments to appear somewhat dark. Under crossed nicols the fragments have a slight pinkish tan color. If the constituent grains are sufficiently coarse, a change from high to low index can be observed on a given grain as the microscope stage is rotated.

15. Glass may develop in the partial fusion of shale (red dog). Glass is amorphous (no crystal structure) and remains dark as the stage is turned under crossed nicols. Glass also has a lower index than most minerals (lower than 1.544) and commonly contains small air bubbles.

3.3.2.3 Chemicals--

1. Acetone (CH_3COCH_3), reagent grade.

2. Dispersing agent: Dissolve 35.7 g sodium metaphosphate (Fisher S-333 or equivalent) and 7.94 g sodium carbonate and dilute to 1 liter with distilled water.

3. 1.544 Index oil (available from R. P. Cargille Laboratories, Inc., Cedar Grove, N.J. 07009 or other suppliers).

3.3.2.4. Materials--

1. Polarized petographic microscope with micrometer stage with 10 X eyepiece and a range in objectives from 3.5 to 50 X.

2. Variable intensity white light source.

3. 7.62 X 2.54 cm (3 x 1 in) glass microscope slide.

4. Slide cover glasses.

5. Thermometer 0-100°C in 1°C divisions.

6. Sieve, 0.25 mm openings (60 mesh).

7. Sieve, 0.177 mm openings (80 mesh).

8. Shaker, horizontal reciprocating type, 6.3 cm (2.5 in) stroke, 120 strokes per minute.

9. Beakers, 250 ml, low-form.

10. Bottles, 950 ml (32 oz dry square).

11. Two beakers, 400 ml, low-form.

12. Two polyethylene bottles, 250 ml.

13. Lens paper.

14. Balance, can be read to 0.1 g.

3.3.2.5 Procedure--

1. Mix bulk field sample thoroughly. CAUTION: Do not use steel utensils to mix sample as some magnetic minerals may be attracted to the iron in the steel.

2. Weigh approximately 100 g of fine-textured (50 g of coarse textured) minesoil.

3. Place weighed sample in a 950 ml (32 oz) dry square bottle.

4. Add 20 ml of dispersing agent.

5. Place bottle on reciprocating shaker. Shake sample overnight.

6. Make a nested series with the 60 mesh and 80 mesh sieves placing the 60 mesh sieve on top.

7. Wet sieve entire sample, being sure to thoroughly wash sample from the bottle.

8. Place sample retained on 80 mesh in a 250 ml beaker. Oven dry sample.

9. Thoroughly mix dried sample. NOTE: See caution step 1.

10. Fill two 250 ml polyethylene bottles with acetone. Label one bottle "Acetone Wash" and the second bottle "Acetone Rinse." Similarly label two 400 ml beakers.

11. Pour acetone from bottle marked "Acetone Wash" into corresponding 400 ml beaker. Repeat for bottle marked "Acetone Rinse."

12. Thoroughly wash and then rinse glass microscope slide and glass slide cover.

13. Air dry glass microscope slide and glass slide cover.

14. Return acetone to appropriate polyethylene bottles. NOTE: The acetone can be used for several wash and rinse cycles. Throw out acetone in "Acetone Wash" bottle when it becomes too contaminated to thoroughly wash slides. Replace discarded acetone from "Acetone Wash" bottle with acetone in "Acetone Rinse" bottle. Put fresh acetone in "Acetone Rinse" bottle.

15. Thoroughly clean glass microscope slide and cover glass with lens paper.

16. Thoroughly clean microscope lens and mirror with lens paper.

17. Place a few grains of the thoroughly mixed oven-dry sample on a glass microscope slide.

18. Add 1.544 index oil by drops until all grains are covered.

19. Place one edge of the cover glass on the microscope slide. Gently lower the opposite edge being careful not to trap air bubbles under the cover glass.

20. Place slide on microscope stage.

21. Adjust white light source, microscope mirror, and microscope diaphragm for best light refraction without being strongly bright.

22. Move stage micrometer to one corner of the cover glass. Note both micrometer readings.

23. While moving stage micrometer in increments of one and doing one row at a time, count various types of grains (see 3.3.2.2) as they appear under the cross hairs until the area under cover glass has been completely covered.

24. Record results for each slide.

25. Thoroughly wash and rinse slide and cover glass (see steps 11-14). Discard grains from bottom of beaker after washing.

3.3.3 Petrographic Analysis of Thin Sections

3.3.3.1 Principle--

Overburdens and minesoils can be studied in slices called thin sections which are 30 microns thick. Thin sections are examined under the petrographic microscope and are useful in observing how individual constituents are arranged and in determining the size and shape of pores. The technique of studying morphological features under the microscope is basically an extension of methods used in studying samples with a hand lens or the unaided eye.

3.3.3.2 Comments--

Coherent samples can be thin sectioned directly but friable samples have to be impregnated usually with a polyester resin (Buol and Fadness, 1961). Normally thin sections are prepared by professionals who advertise in several geological and mineralogical journals and magazines.

Thin sections should first be examined with low magnification to study larger scale features and familiarize the observer with the minerals and fabrics within the thin section. Magnification is increased to observe finer details. Observation with transmitted light is conducted in plain light, crossed polarized light with and without substage condensing lens, and with light stopped down to different degrees. Some features may be more visible in reflected light. The determination of approximate percentage of constituents can be ascertained by visual estimation. More accurate determinations can be made by counting the different kinds of grains, voids and special features.

The mineral composition of the larger grains (skeletal grains) observed in thin section can be determined by procedures similar to those used in the immersion method (see 3.3.2.2). The larger grains are generally embedded in a matrix of very fine-grained material composed largely of fine silt and clay. Some of the matrix material may be identified (see 3.3.2.2) but complete identifications require x-ray diffraction and differential thermal analysis.

More information on identification of minerals in thin sections may be obtained from Cady (1965) and Kerr (1959); however, the following special features may be seen in thin sections:

1. Cutan is a general term coined by Brewer (1964) to designate accumulations on soil particle surfaces or textural changes along a surface of movement. Accumulations may be composed of various materials such as clay, organic matter, silica, iron oxides or hydroxides, or manganese oxides or hydroxides.

2. Argillans or clay skins are cutans composed of clay minerals and occur on the natural surfaces of soil particles. They have a smooth or ropy surface with a waxy luster in reflected light. In thin sections they have the same index as the clay matrix but a higher index than that of quartz or feldspar grains. Under crossed nicols argillans commonly appear as white borders on grains or soil units. The borders go to extinction at the points where they are parallel to the polarizing elements ("north-south and east-west").

3. Ferrans are iron oxides or hydroxides occurring on soil surfaces. In thin sections they are translucent or opaque. Under reflected light they are yellow, brown, or red.

4. Mangans are manganese oxides or hydroxides which are usually opaque and very dark brown or black in reflected light.

5. Concretions differ in composition from the material which surrounds them. Concretions may be composed of any mineral matter, but they are more commonly made up of carbonate, chert, sulfate, or oxides and hydroxides of iron or manganese. Normal technqiues of mineral identification are used to distinguish between the different types of concretions.

6. Earthworm casts appear as tubes of material commonly containing rounded aggregates representing excreta. The aggregates have dark outer borders of humus and may occur in clusters.

7. Root channels are voids left from decayed roots. These voids resemble worm burrows but may contain remnants of roots with characteristic cellular structure. The channel system may also show more of a tree-like pattern than do the worm burrows.

8. Pores (voids) in thin sections are colorless in plain light and remain black when the stage is rotated under crossed nicols. The percentage of observed pores can be estimated by visual inspection or determined more accurately by the point count method as described by Anderson and Binnie (1961).

The shape of the pore may be spherical, tubular, planar, or irregular. Spherical voids are commonly referred to as vesicles, some of which result from gas bubbles or solution of spherical grains. The apparent shape of the other pores depends on how they are cut during thin section preparation. Tubular pores will appear round in sections cut at right angles to the tubes. The shape will be elliptical and more elongate the more nearly the section parallels the length of the tubes. Tubular pores commonly result from burrowing by earthworms and insects or from the decay of plant roots. The cut of the section is not as critical in recognizing planar voids, although the true width of the opening can be determined only on sections at right angles to the plane of the voids. The planar voids commonly originate as a result of shrinkage of soil material as it dries. Our data show that soil fabrics, as described by Brewer (1964), do not normally occur in young minesoils. Remnant soil fabrics may be seen in minesoils that have been "top soiled" or mixed with a natural soil.

More details on soil fabrics can be found in Brewer (1964). Sampling procedures and number of grains to be counted are given by Cady (1965), Kerr (1959), and Winchell (1937). Accurate percentage determinations by point counting methods are given by Hutchison (1974) and Anderson and Binnie (1961).

3.3.3.3 Chemicals--

None required.

3.3.3.4 Materials--

1. Polarized petrographic microscope with micrometer stage with 10 X eyepiece and a range of objectives from 3.5 to 50 X.

2. Variable intensity white light source.

3.3.3.5 Procedure--

1. Place thin section on the stage of the petrographic microscope.

2. Adjust light intensity so grains can be easily seen.

3. Examine thin section under low magnification. Use higher magnification to clarify details.

4. Determine kinds of voids and percent porosity.

5. Examine for oriented clay bodies and determine position and percentage in sample.

6. Determine mineralogy and percentage of the skeletal grains.

7. Examine for special features (see 3.3.2.2) and record percentage of each.

3.3.4 Identification of Clay Minerals by X-Ray Diffraction

3.3.4.1 Principle--

The clay minerals of greatest interest (e.g. kaolinite, illite (mica), vermiculite, chlorite, and montmorillonite) are mostly flaky or platy in shape. They are readily identified and distinguished from one another by observing the effect of different chemical and heat treatments on the interlayer spacings along the axis perpendicular to the platy surfaces with the use of x-ray diffraction.

The pretreatment used to distinguish montmorillonite from vermiculite and chlorite and to identify illites is saturation of the exchange complex of the clay with magnesium and treatment with glycerol. Vermiculite is distinguished from chlorite and kaolinite by saturating the clays with potassium and heating on a glass slide at 500°C. Intermediate heat treatments, 110°C and 250°C, can be used to study interlayering in the collapsing minerals or other special problems. Stronger x-ray diffraction peak intensities are obtained due to preferred orientation of the clays on the glass slide. This preferred orientation results since the clay plates settle parallel or nearly parallel upon drying from the suspension.

3.3.4.2 Comments--

Due to the length of time involved in sample preparation, several samples should be prepared at the same time. The commonly used radiation sources are copper and cobalt. If copper radiation is used, free iron oxides will have to be removed (see Jackson, 1958 p. 168) to eliminate interference. Interpretation of data should be performed by a person qualified in x-ray analysis and clay mineralogy.

3.3.4.3 Chemicals--

1. Sodium hydroxide (NaOH), 1 N: Dissolve 40.0 g of NaOH pellets with carbon dioxide-free water (See 3.2.3.3 No. 1) and dilute to a volume of 1 liter. Protect from CO_2 in air with ascarite tube.

2. Sodium carbonate (Na_2CO_3), 1 N: Dissolve 53 g of Na_2CO_3 with carbon dioxide-free water (See 3.2.3.3 No. 1) and dilute to a volume of 1 liter. Protect from CO_2 in air with ascarite in a guard tube.

3. pH 10 water: Dilute 10 ml of 1.0 N Na_2CO_3 to 10 liters with distilled and deionized water. Check pH with a pH meter and adjust to pH 10 by the addition of 0.1 N HCl or 1.0 N Na_2CO_3.

4. Acetone (CH_3COCH_3).

5. Hydrochloric acid (HCl), 1.0 N: Dilute 83 ml of concentrated HCl to a volume of 1 liter with distilled water.

6. Hydrocholoric acid (HCl), 0.1 N: Dilute 100 ml of 1 N HCl to a volume of 1 liter with distilled water.

7. Bromophenol Blue.

8. Hydrogen peroxide (H_2O_2), 30%, ACS certified (without added preservative).

9. Sodium acetate ($NaC_2H_3O_2$), 1 N: Dissolve 82 g of $NaC_2H_3O_2$ with distilled water and dilute to a volume of 1 liter. Buffer to pH 5.0 with acetic acid or sodium hydroxide.

10. Magnesium chloride ($MgCl_2 \cdot 6H_2O$), 1 N: Dissolve 102 g of $MgCl_2 \cdot 6H_2O$ with distilled water and dilute to a volume of 1 liter.

11. Magnesium chloride ($MgCl_2 \cdot 6H_2O$), 10 N: Dissolve 1020 g of $MgCl_2 \cdot 6H_2O$ with distilled water and dilute to a volume of 1 liter.

12. Potassium chloride (KCl), 1 N: Dissolve 74.5 g of KCl with distilled water and dilute to a volume of 1 liter.

13. Potassium chloride (KCl), 10 N: Dissolve 745 g of KCl with distilled water and dilute to a volume of 1 liter.

14. Magnesium acetate ($Mg(C_2H_3O_2) \cdot 4H_2O$), 1 N: Dissolve 107 g of $Mg(C_2H_3O_2) \cdot 4H_2O$ with distilled water and dilute to a volume of 1 liter.

15. Potassium acetate (CH_3COOK), 1 N: Dissolve 98 g of CH_3COOK with distilled water and dilute to a volume of 1 liter.

16. Methanol (CH_3OH).

17. Silver nitrate $AgNO_3$, 10% : Dissolve 10 g of $AgNO_3$ with distilled water and dilute to a volume of 100 ml.

18. Glycerol solution, 20% : Dilute and mix 20 ml of glycerine ($CH_2OHCHOHCH_2OH$) to 100 ml with distilled water.

3.3.4.4 Materials--

1. Balance, can be read to 0.1 g.

2. Soil dispersion mixer with baffled cup.

3. Sieve, 300 mesh.

4. Rubber policeman.

5. Funnel, large powder, polyethylene.

6. Beakers, 1000 ml, 600 ml, and 400 ml.

7. Drying oven.

8. Spatula.

9. Spot plate.

10. Hot plate.

11. Centrifuge bottles, 250 ml with screw caps and centrifuge tubes, 50 ml.

12. Centrifuge; equipped with tachometer and timer (IEC Model K with No. 277 and 279 heads or equivalent centrifuge with 4-place and 12-place heads).

13. Bottle, French square, 1 liter (32 oz) capacity.

14. Shaker, horizontal reciprocating type, 6.3 cm (2.5 in) stroke, 120 strokes per minute.

15. Flask, Florence, 2000 ml with rubber stopper.

16. Vacuum desiccator.

17. X-ray diffraction instrument.

18. Glass x-ray slides.

19. Vortex mixer.

3.3.4.5 Procedure--

NOTE: If soil samples are analyzed instead of rock samples, omit 3.3.4.5.1.

3.3.4.5.1 Separation of clay from rock samples -- The following steps are for the separation of clay from rock samples only.

1. Weigh 100 g of ground (less than 60 mesh) rock material in a 600 ml beaker.

2. Add 300 ml distilled water.

3. Adjust the pH to 8.5 with 1.0 N NaOH.

4. Transfer suspension to metal container used with the soil dispersion mixer.

5. Fill to two-thirds of the container's volume with pH 10 water.

6. Stir suspension vigorously (using soil dispersion mixer) for 30 to 60 minutes.

7. Pour most of the suspension (all except heavy soup near bottom of container) into a 300 mesh sieve, aiding passage of the suspension through the sieve with a gentle jet of pH 10 water. NOTE: Sieve should be mounted in a large polyethylene funnel leading into a 1000 ml beaker.

8. Wash residue remaining on sieve with a gentle jet of pH 10 water, gently breaking up any clay or silt lumps with a rubber policeman.

9. Add pH 10 water to residue in the metal container and repeat steps 5 through 8.

10. Repeat step 9 until most of the silt and clay has been washed from the container. Transfer the sand in the container to the sieve and wash several times with a jet of pH 10 water, being certain to break up the lumps of silt and clay with a rubber policeman.

11. Wash sand with acetone to remove most of the water.

12. Dry the sand. After drying, shake sieve for about 10 minutes either by hand or mechanical shaker. Add material that passes sieve to the 1000 ml beaker containing silt and clay fractions.

13. Carefully pour contents of the sieve onto a black glazed paper, turning the sieve over and tapping its rim with the handle of a spatula.

14. Weigh sand and store in vial if analysis of the sand is desired. If sand is not needed for analysis, discard after weighing.

15. Go to 3.3.4.5.2 step 18.

3.3.4.5.2 Separation of soil fractions -- The following steps are for separation of soil fractions only.

1. Weigh 50 g of soil and transfer to a 1000 ml beaker.

2. Add 150 ml deionized water.

3. Adjust to pH 3.5 with 1.0 N HCl using bromophenol blue indicator and a spot plate. So as not to lose any soil, wash the drops of suspension on the spot plate back into the original beaker.

4. Add 25 ml of 30% H_2O_2 and cover with a watch glass. Allow to stand overnight without heating.

5. The following day, add an additional 25 ml of 30% H_2O_2 and heat gently to 90°C on a hot plate. Continue heating for at least one hour maintaining the sample at 90°C. NOTE: If the soil contains a large amount of organic matter, add an additional 25 ml of H_2O_2 after 1 to 2 hours and continue to repeat the additions until most of the organic "scum" is destroyed (this may take all day with 3 or 4 applications of H_2O_2).

6. Wash soil into a 250 ml centrifuge bottle, adjust to pH 3.5 if necessary, and centrifuge at 1,500 RPM until the supernatant liquid is clear (about 5 minutes).

7. Discard the clear liquid, transfer soil to a 400 ml beaker and adjust to pH 10 using a pH meter and 1.0 N Na_2CO_3 solution.

8. Pour suspension into a 1 liter bottle and fill to two-thirds of volume with pH 10 water.

9. Place horizontally on a reciprocating shaker and shake for 16 hours at 120 strokes per minute.

10. Place a 300 mesh sieve in a large polyethylene funnel and place funnel over a 1000 ml beaker.

11. Pour most of the suspension from the bottle into the sieve, washing silt and clay through sieve with a gentle jet of pH 10 water.

12. Wash remaining material from the bottle onto the sieve with a gentle jet of pH 10 water.

13. Wash silt and clay through sieve with a jet of pH 10 water, breaking silt and clay lumps with a rubber policeman.

14. Wash remaining sand with acetone to remove most of the water.

15. Dry sand. Add a sieve cover and receiving pan to the sieve and vigorously shake either by hand or mechanical shaker for 10 minutes. Material passing sieve is added to the 1000 ml beaker containing silt and clay fractions.

16. Carefully pour the contents of the sieve onto black glazed paper, turning the sieve over and tapping its rim with the handle of a spatula.

17. Weigh sand and transfer to storage vial. If sand is not needed for analysis, discard after weighing.

18. Pour contents of the 1000 ml beaker into a 250 ml centrigute bottle, using one bottle per sample. Balance bottles. Centrifuge at 2000 RPM for 5 minutes. Pour supernatant suspension into a 2000 ml Florence flask labeled less than 2 micron fraction. NOTE: Since all of the suspension in the 1000 ml beakers will not fit into the 250 ml centrifuge bottle, centrifuging must be repeated by making additions to the bottle after each decantation until all of the suspension in the 1000 ml beaker is centrifuged. Do not stir material in the bottom of the centrifuge bottle between these additions.

19. After all of the suspension in the 1000 ml beaker has been added and centrifuged, add pH 10 water to the bottles, stir, and centrifuge at 1500 RPM for 10 minutes. Decant supernatant liquid into the Florence flask.

20. Add pH 10 water, stir, and this time centrifuge at 1600 RPM for exactly 2 minutes. Decant into the Florence flask.

21. Repeat step 20 until the supernatant liquid is about clear. NOTE: If the Florence flask becomes filled, an auxiliary container such as a 2000 ml beaker can be used. Both the Florence flask and the auxiliary container must be kept covered with a stopper or watch glass.

22. Add 20 ml of 1 N $NaC_2H_3O_2$ to flocculate the clay. After clay flocculates, siphon off as much of the liquid as possible without removing any of the clay.

23. Stir remaining liquid and clay by hand and pour mixture into a beaker.

24. Wash remaining clay from the Florence flask into the beaker with deionized water.

25. Place beaker in a vacuum desiccator, attach vacuum line, and evaculate until clay is air-dry.

3.3.4.5.3 Mg (or K) saturation of clay fraction -- The following steps include procedures for either Mg or K saturation of clays depending on analyses required.

1. Weigh 0.10 g of air-dry clay and suspend in 100 ml of 1 N $NaC_2H_3O_2$ (buffered to pH 5.0) in a 250 ml centrifuge bottle. Boil gently for 5 minutes.

2. Add 20 ml of 1 N $MgCl_2 \cdot 6H_2O$ (or KCl for samples to be K saturated) to the suspension.

3. Mix the suspension thoroughly and centrifuge at 2000 RPM for 5 minutes.

4. If the supernatant liquid is clear, discard the liquid; if not, add 10 ml of 10 N $MgCl_2 \cdot 6H_2O$ (or KCl) to insure flocculation. Centrifuge and discard clear supernatant liquid.

5. Wash clay once with 1 N $Mg(C_2H_3O_2) \cdot 4H_2O$ (or CH_3COOK) and twice with 1 N $MgCl_2 \cdot 6H_2O$ (or KCl) to remove acetates centrifuging and discarding clear supernatant liquid between washings.

6. Wash and centrifuge clay twice with 20 ml deionized water and then with methanol until free from chlorides (Cl^- is present if a precipitate occurs with the addition of a few drops of 10% $AgNO_3$ to a few ml. of the supernatant.

7. Pipet about half the suspension. Spread suspension on a glass slide and allow to dry at room temperature (25°C)(see 3.3.4.5.5, step 2).

8. Dry remaining clay over drierite in a desiccator at room temperature (25°C) and proceed directly to glycerol solvation (3.3.4.5.4) if glycerol treatment is needed; if not, proceed to 3.3.4.5.5.

3.3.4.5.4 Glycerol solvation -- The following steps are necessary if glycerol saturation is required.

1. Weigh 0.050 g air-dry clay (Mg saturated) and place in a 50 ml centrifuge tube.

2. Add 0.5 ml of 20% glycerol solution.

3. Let stand for 30 minutes, centrifuge, and let drain for at least 30 minutes.

4. Add about 1 ml of water to the centrifuge tube to make a free flowing slurry and mix using a Vortex mixer.

3.3.4.5.5 Slide preparation and treatment selection -- From the following steps, x-ray slides can be prepared and proper treatments chosen.

1. Select appropriate treatments and temperatures from Tables 9 and 10. NOTE: All heat treatments are heated at the given temperatures for 2 hours.

2. Carefully pipet 1 or 2 ml suspension onto glass slide. Do not allow suspension to run off the glass slide, but cover an area large enough to cover the entire x-ray beam.

3. Dry at room temperature (25°C).

4. Run x-ray analysis following manual supplied with the x-ray diffraction equipment.

5. Perform appropriate treatments as determined in 3.3.4.5.5, step 1. Rerun slides on x-ray diffraction unit.

6. Determine basal spacing in angstroms (Å) depending on type of radiation used.

7. Determine type of clay minerals using data from Tables 9 and 10.

3.3.4.5.6 Explanatory notes -- The following notes are for Tables 9 and 10.

1. Peaks sharp and higher orders distinct; beidellite closes easier on K saturation than montmorillonite from bentonite.

2. Peaks broad; higher orders very weak.

3. Peaks broader than mica; higher orders not quite as distinct and may not be exactly integral; may be interstratified with montmorillonite; 10 Å peak sharpened on heating clay.

4. First order peak sharper than montmorillonite but not as sharp as mica; higher orders are weak.

5. Strong 2nd order reflection; peaks sharp; spacing may vary ± 0.2 Å.

TABLE 9. BASAL SPACINGS OF CLAY MINERALS AS INFLUENCED BY Mg-SATURATION AND GLYCEROL TREATMENTS

	Basal Spacings (Å units)		
Mineral Name	H_2O 25°C	glyc 25°C	Notes (see 3.3.4.5.6)
Mica	10	10	1
Montmorillonite (mont.)	12-15	18	2, 10
Illite	10	10-11	3, 10
Vermiculite (verm.)	14.5	14.7	4, 10
Chlorite	14.4	14.4	5, 10
Interstratified mont. and illite	11-14	15-17	6, 10
Interstratified verm. and chlorite	14.5	14.6	6, 10
Interstratified verm. and illite	11-14	11-14	6, 10
Montmorillonite with "interlayer islands"	14-15	14-15	7, 10
Vermiculite with "interlayer islands"	14-15	14-15	7, 10
Interstratified mont. with "islands" and illite	11-14	11-14	7, 10
Interstratified verm. with "islands" and illite	11-14	11-14	7, 10
Kaolinite	7.0-7.2	7.0-7.2	8, 10
Hydrated halloysite	10	10	8, 9
Dehydrated halloysite	7.2	7.2	9

TABLE 10. BASAL SPACINGS OF CLAY MINERALS AS INFLUENCED BY K-SATURATION AND HEAT TREATMENTS

	Basal Spacing (Å units)				Notes
Mineral Name	25°C	100°C	300°C	525°C (see	3.3.4.5.6)
Mica	10	10	10	10	1
Montmorillonite (mont.)	10-12	10	10	10	2, 10
Illite	10	10	10	10	3, 10
Vermiculite (verm.)	10.5	10.2	10.1	10.1	4, 10
Chlorite	14.4	14.4	14.4	14.4	5, 10
Interstratified mont. and illite	10-11	10	10	10	6, 10
Interstratified verm. and chlorite	11-13	11-13	11-13	11-13	6, 10
Interstratified verm. and illite	10-11	10-11	10-11	10	6, 10
Montmorillonite with "interlayer islands"	14	13-14	11-12	10-11	7, 10
Vermiculite with "interlayer islands"	11-14	11-14	11-12	10-11	7, 10
Interstratified mont. with "islands" and illite	11-14	11-14	11-13	10	7, 10
Interstratified verm. with "islands" and illite	11-14	11-13	11-13	10	7, 10
Kaolinite	7.0-7.2	7.0-7.2	7.0-7.2	None	8, 10
Hydrated halloysite	10	7.2	7.2	None	8, 9
Dehydrated halloysite	7.2	7.2	7.2	None	9

6. Higher orders not integral.

7. Peaks may not sharpen on heating but shift to smaller "d" values.

8. Well crystallized kaolinite has a sharp peak at 7.0 Å and higher orders are present; poorly crystallized kaolinite has a broader peak at 7.2 and may be confused with halloysite.

9. If hydrated halloysite is once dried in the absence of some excess salts it does not reexpand to 10 Å. If dried in a slurry of HCl or NH_4Cl the spacing remains at 10 Å.

10. All 2:1 minerals are probably interstratified or interlayered with "islands" of nonexchangeable groups to some degree. When it is slight there is some shifting of the spacing indicated. The greater the proportion of a particular phase, the more the spacing will be like that of the pure mineral.

3.4 PHYSICAL METHODS

3.4.1 Summary--

The methods listed in this section are primarily for minesoil and soil materials. These methods measure parameters that dictate the long-term use of the soil. Where chemical properties are of extreme importance in the short term, physical properties of minesoils are extremely important to long-term management and use. Chemical properties can be more easily modified and changed than physical properties.

The size distribution of particles can be measured by either of the two methods, pipette or hydrometer. The pipette (3.4.2) is the more exact while the hydrometer method (3.4.3) is less time consuming. Bulk density can be measured by methods 3.4.4 through 3.4.7. The type of materials found in minesoils with the large variety of particle sizes dictated that more than one method be presented to the user for measuring bulk density. The other methods are self-explanatory and need no further clarification; however, material used in each of the physical methods is only sieved when taken from the field and not subjected to grinding.

3.4.2 Particle Size Analysis (Pipette Method)

3.4.2.1 Principle --

The pipette method depends on differential settling rates of silt- and clay-size soil particles from a water suspension. Since large particles settle faster than small particles of similiar density (as stated by Stokes' Law), sampling a suspension at constant depth over increasing longer periods of time will yield increasingly smaller sizes of the suspended solids. By sampling the suspension with a pipette at a 10 cm depth at the time calculated from Stokes' Law, a sample of specific equivalent particle sizes will have already settled past the 10 cm depth at each sampling time.

3.4.2.2 Comments —

Normally soil samples are pretreated with hydrogen peroxide to remove organic matter. Materials which contain concentrations of soluble salts and gypsum must be leached with enough water to remove them before good dispersion of the sample can be accomplished. If the need should arise, procedures for the removal of organic matter, soluble salts, and gypsum are available (Day, 1956; Kilmer and Alexander, 1949).

3.4.2.3 Chemicals --

Dispersing agent: Dissolve 34.7 g sodium metaphosphate ($Na(PO_3)_6$) (Fisher Scientific Co. No. S-333 or equivalent) and 7.94 g sodium carbonate (Na_2CO_3) in distilled water and dilute to one liter. The Na_2CO_3 is used as an alkaline buffer to prevent the hydrolysis of the metaphosphate back to the orthophosphate which occurs in acidic solutions. NOTE: Instant Calgon available from Calgon Corp., Pittsburgh, Pa. can be used.

3.4.2.4 Materials--

1. Sieve, 2 mm (10 mesh) openings.

2. Sieve, approximately .05 mm (300 mesh) openings.

3. Bottles, pyrex nursing, 237 ml (8 oz) with rubber stoppers.

4. Pipette, Lowry automatic, 25 ml (available from Arthur H. Thomas, Co., Philadelphia, Pa.).

5. Shaw pipette rack. NOTE: If not available, a substitute pipette rack can be made using a cathetometer or a support stand with a sliding clamp. The pipette rack is required for lowering and positioning the tip of the pipette at a controlled depth below the upper mark of the 1000 ml graduated cylinder.

6. Balance, can be read to 0.001 g.

7. Drying oven.

8. Hot plate

9. Wooden rolling pin.

10. Beakers, 250 ml.

11. Watch glass.

12. Desiccator with drierite desiccant.

13. Funnel.

14. Rubber policeman.

15. Shaker, horizontal reciprocating type, 6.3 cm (2.5 in) stroke, 120 strokes per minute.

16. Cylinders with a 1000 ml graduation (KIMAX brand (20023) or equivalent).

17. Aluminum pan.

18. Plunger (see 3.4.3.4 No. 6).

19. Vacuum assembly, vacuum source and suction hose with valves and trap to control rate (Kilmer and Mullins 1954, Figure 6, p. 440).

20. Weighing bottles, 60 ml capacity or 100 ml beakers.

3.4.2.5 Procedure (Adapted from Kilmer and Alexander, 1949)--

1. Mix and quarter air-dry soil.

2. Roll one quarter with a wooden rolling pin to break up clods.

3. Pass sample through 2 mm sieve. NOTE: Rolling and sieving are repeated until only rock fragments and pebbles are retained on the sieve. CAUTION: Care must be taken to avoid breaking the rock fragments.

4. Material not passing the 2 mm sieve are weighed and reported as a percentage of the air-dry weight of the whole sample.

5. Two 10.000 g samples of air-dry material passing the 2 mm sieve are weighed.

6. One sample is placed in a weighing bottle and dried at 105°C overnight. Then it is cooled in a dessicator and weighed to the nearest milligram. This weight is recorded as organic-free, oven-dry weight.

7. The other 10.000 g subsample is placed in a 237 ml (8 oz) nurse bottle with 10 ml of the dispersing agent.

8. Add distilled water to bring volume to about 177 ml (6 oz.). Stopper bottle and shake overnight in a horizontal position on a reciprocating shaker at 120 strokes per minute.

9. After shaking, bring bottle to room temperature by allowing it to stand for a few minutes if necessary.

10. Place 300 mesh sieve in a funnel and then place funnel in a 1000 ml graduated cylinder.

11. Wash the dispersed sample on the 300 mesh sieve. Wash all sample from the bottle using a jet of distilled water. CAUTION: Jets of water should be avoided in washing the sample through the sieve. The funnel should be gently tapped with the side of the hand to facilitate the washing procedure. Care should be taken not to spill any material over the top of the sieve.

12. Continue washing until the volume in the cylinder totals about 500 ml. NOTE: Sands remain on the sieve. It is necessary that all particles of less than about 50 microns diameter be washed through the sieve.

13. Remove sieve, place in a tarred aluminum pan and dry in the oven. Cool sieve and transfer sands to the pan using a brush. Dry the pan and contents for about 2 hours at 105°C. The pan is then placed in a desiccator to cool and the contents weighed to the nearest 0.01 g.

14. Wash materials retained in the funnel into the cylinder. Bring volume to 1000 ml graduation mark with distilled water.

15. Cover cylinder with a watch glass and set in sedimentation cabinet.

16. Place the Lowry pipette on the pipette rack.

17. Make adjustments required to immerse the pipette 10 cm in the suspension when proper sampling time has arrived.

18. Attach vacuum line to pipette and adjust vacuum assembly to fill pipette in 12 sec using distilled water.

19. Stir the material in the sedimentation cylinder for 6 minutes (8 minutes if the suspension has stood for more than 16 hours) with a motor driven stirrer. CAUTION: Do not let any of the suspension spill over the top of the cylinder.

20. After mechanical stirring, stir the sample using an up and down motion for 2 minutes with the plunger (see 3.4.3.4 No. 6). Record time at completion of stirring and suspension temperature. NOTE: The temperature should remain constant during the settling process by using a constant temperature room or placing cylinders in a constant temperature bath. Samples should be placed where they are free of vibrations.

21. Using Table 11, determine the settling time for the less than 20 micron fraction.

22. About one minute before the determined settling time, the tip of the pipette is lowered slowly into the suspension to a depth of 10 cm by means of the pipette rack.

23. At the appropriate time, fill the pipette by controlled suction calibrated to require 12 seconds to fill. Remove pipette. Drain freely into a pre-weighed weighing bottle or beaker.

24. Add one rinse from the pipette to the weighing bottle or beaker using distilled water.

25. Repeat steps 22 through 24 until all cylinders have been sampled for the less than 20 micron fraction.

26. Restir samples for 2 minutes using an up and down motion with the plunger.

TABLE 11. TIMES FOR PARTICLE SIZE ANALYSIS (PIPETTE METHOD) BASED ON TEMPERATURE

°C	Less Than 20 Micron	Less Than 2 Micron
20°	4 min. 40 sec.	466 min. = 7 hr. 46 min.
21°	4 min. 33 sec.	455 min. = 7 hr. 35 min.
22°	4 min. 27 sec.	444 min. = 7 hr. 24 min.
23°	4 min. 20 sec.	434 min. = 7 hr. 14 min.
24°	4 min. 14 sec.	424 min. = 7 hr. 4 min.
25°	4 min. 9 sec.	415 min. = 6 hr. 55 min.
26°	4 min. 3 sec.	405 min. = 6 hr. 45 min.
27°	3 min. 58 sec.	396 min. = 6 hr. 36 min.
28°	3 min. 53 sec.	388 min. = 6 hr. 28 min.
29°	3 min. 48 sec.	379 min. = 6 hr. 19 min.
30°	3 min. 43 sec.	372 min. = 6 hr. 12 min.

27. Record time and temperature.

28. Using Table 11, determine the settling time for the less than 2 micron fraction.

29. Repeat steps 22 through 24 until all cylinders are sampled.

30. Dry weighing bottles or beakers in an oven at 90°C until the volume has been reduced by one-half. Then dry for 12 hours at 105°C.

31. Cool in desiccator and record weights of the individual fractions.

32. Prepare a blank by placing 10 ml of the dispersing agent in a 1000 ml graduated cylinder. Bring volume to 1000 ml with distilled water. Pipette 25 ml and place in preweighed weighing bottle or beaker along with one rinse of the pipette. Dry at 105°C, cool in desiccator, and record weight as weight correction factor for dispersing agent.

3.4.2.6 Calculations--

1. % sand = (Weight of sand fraction/Weight of oven-dry, organic-free total sample) X 100.

2. Constant (K) = 1000/Volume of pipette.

3. D = 100/Weight oven-dry, organic-free total sample.

4. % Clay = (A - B) KD, where:

A = Weight in grams of the less than 2 micron fraction plus dispersing agent.

B = Weight in grams of dispersing agent correction.

5. % (20 to 2 micron) = [(A - B)KD] - (% clay), where:

A = Weight in grams of the less than 20 micron fraction plus dispersing agent.

B = Weight in grams of dispersing agent correction.

6. % (50 to 20 micron) = 100 - [% sand + % clay + % (20 to 2 micron)].

7. % silt = (% 20 to 2 micron) + (% 50 to 20 micron).

3.4.3 Particle Size Analysis (Hydrometer Method)

3.4.3.1 Principle--

This method depends on the rate at which soil particles settle from a water suspension. The soil particles are put into suspension by mechanical stirring with the aid of a dispersing agent. Sodium metaphosphate solution is used to disperse the soil and avoid flocculation of the clays. Sodium replaces exchangeable calcium and the precipitation of the calcium, in the form of calcium phosphate, prevents its recombination with the clays. The net negative charge on the clay particles increases due to the addition of sodium ions, causing the particles to repel each other and disperse. Since large partilces settle faster than the same kind of small particles as stated by Stokes' Law, the concentration of soil particles in suspension at a given time is dependent upon the size of the particles.

3.4.3.2 Comments--

Temperature is important in the sedimentation procedure since the density and viscosity of water change with temperature. As the temperature increases, the time required for particles to settle out of suspension decreases. The hydrometer is usually calibrated for 19.4 or 20°C (67 or 68°F). For each °F above the hydrometer calibration temperature, add 0.2 g to the reading. Subtract 0.2 g from the hydrometer reading for each °F below the calibration temperature. Although the correction factor for temperature can be used, it is best to carry out the procedure in a constant temperature room or maintain the sedimentation cylinders in a constant temperature bath.

The material is mixed using a reciprocating shaker; however, a soil dispersion mixer with baffled cup (similar to a drink mixer) can be used as an alternate method if a reciprocating shaker is not available. With this apparatus, a weighed sample is placed in the baffled cup with distilled water and dispersing agent. The cup is placed on the mixer and stirred for a maximum of 5 minutes. Many minesoil samples can usually be mixed in 3 minutes.

3.4.3.3 Chemicals--

1. Dispersing agent: Dissolve 35.7 g sodium metaphosphate ($Na(PO_3)_6$) (Fisher Scientific Co. No. S-333 or equivalent) and 7.94 g sodium carbonate (Na_2CO_3) in distilled water and dilute to a volume of 1 liter. The Na_2CO_3 is used as an alkaline buffer to prevent the hydrolysis of the metaphosphate back to orthophosphate which occurs in acidic solutions. NOTE: Instant Calgon available from Calgon Corp., Pittsburgh, Pa. can be substituted.

2. Distilled water.

3.4.3.4 Materials--

1. Bottles, French square, 1 liter (32 oz) with caps.

2. Shaker, horizontal reciprocating type, 6.3 cm (2.5 in) stroke, 120 strokes per minute.

3. Glass sedimentation cylinder with markings at the 1130 ml and 1205 ml levels (Bouyoucos cylinder).

4. Standard hydrometer (ASTM 152 H, with Bouyoucos scale in grams per liter).

5. Balance, can be read to 0.1 g.

6. Plunger. NOTE: This can be made using 3 mm (0.125 in) diameter wire. At one end make a circle 5.5 cm (2.125 in) in diameter. The wire should be manipulated so the handle extends at a right angle from the center of the circle for 56 cm (22 in). Stretched rubber bands bisecting the wire circle are spaced around the circumference until it is largely covered by rubber bands overlapping at the center.

7. Thermometer, 0-100°F.

3.4.3.5 Procedure (Modified from Bouyoucos, 1951)--

1. Weigh 50 g (oven-dried at 105°C overnight) of a fine textured or 100 g of coarse textured (90-100% sand) soil and place in a shaker bottle.

2. Add 125 ml of dispersing agent and 400 ml of distilled water to shaker bottle.

3. Cap bottle snugly and place horizontally on a reciprocating shaker for 16 hours at 120 strokes per minute.

4. Remove bottle and bring suspension to room temperature.

5. Wash all contents of shaker bottle into a sedimentation cylinder.

6. Set cylinder in a place away from vibrations.

7. Place hydrometer in suspension.

8. Fill to lower mark (1130 ml) with distilled water for a 50 g sample. Fill to upper mark (1205 ml) for a 100 g sample.

9. Remove hydrometer. Take plunger in one hand holding the cylinder with the other. Strongly move plunger up and down being careful not to spill contents of cylinder.

10. After all sediment is off cylinder bottom, carefully remove plunger and record time immediately. NOTE: Add a drop of amyl alcohol if the surface is covered with foam and restir the suspension if necessary.

11. Record hydrometer reading at meniscus top at the end of 40 seconds. NOTE: About 10 seconds before taking reading, carefully insert hydrometer and steady by hand.

12. Remove hydrometer from suspension. CAUTION: Do not leave hydrometer in suspension longer than 20 seconds as particles will settle out on its shoulders.

13. Measure and record suspension temperature. For each °F above the calibrated temperature of the hydrometer add 0.2 g to the reading. For each °F below the calibrated temperature subtract 0.2 g.

14. Record corrected hydrometer reading.

15. With the plunger, restir suspension. Take a reading at the end of two hours. Correct hydrometer reading (see step 13) and record corrected hydrometer reading.

16. Make 3 blanks by placing 125 ml of dispersing agent in 3 sedimentation cylinders. NOTE: Blanks should be run for each new batch of dispersing agent.

17. Fill cylinders two-thirds full with distilled water. Insert hydrometer and fill cylinder to the lower mark (1130 ml) with distilled water.

18. Take hydrometer reading and temperature of suspension. Correct hydrometer reading using step 13.

3.4.3.6 Calculations--

1. Dispersing agent correction factor = Sum total of temperature corrected hydrometer readings of blanks/3.

2. Weight corrected 2 hour reading = (Temperature corrected 2 hour hydrometer reading) - (Dispersing agent correction factor).

3. Weight corrected 40 second reading = (Temp. corrected 40 second hydrometer reading) - (Dispersing agent correction factor).

4. % Clay = (Weight corrected 2 hour reading/oven-dry weight of total sample) X 100.

5. % Silt = [(Weight corrected 40 second reading - Weight corrected 2 hour reading)/oven-dry weight of total sample] X 100.

6. % Sand = 100 - (% clay + % silt).

3.4.4 Bulk Density (Core Method)

3.4.4.1 Principles--

The soil bulk density determination is based on two measurements, a mass measurement and a volume measurement. The mass is measured by oven drying the sample at 105°C until a constant weight is obtained. The bulk volume measurement includes the space between the soil particles as well as the space occupied by the soil particles. Bulk density, the ratio of sample mass to sample volume, is expressed as grams per cubic centimeter (Blake, 1965).

3.4.4.2 Comments--

This method may be difficult or impractical in soil containing many rock fragments.

A flat soil surface is prepared at the desired depth and the core sampler is driven into the soil. If driven with a heavy hammer, the head of the tool must be protected with a tough wooden plank or block. Care must be taken to see that no compaction takes place so that a known volume of soil is obtained. The sample is transferred to the laboratory and weighed while still moist. The sample is then dried in an oven and weighed again. This sample must be immediately placed in a desiccator after removing from the oven as the dry sample will absorb moisture from the atmosphere (Baver, 1956, p. 180-182).

3.4.4.3 Chemicals--

None required.

3.4.4.4 Materials--

1. Double-cylinder core sampler with steel cutting edge, driving head,

and removable brass or aluminum sleeves.

2. Core cylinder, 7.6 cm (3 in) in diameter and 7.6 cm (3 in) in height with 3.2 mm (0.125 in) thick walls.

3. Balance, can be read to 0.1 g.

4. Drying oven.

5. One-pint containers.

6. Air tight plastic bags.

7. Aluminum weighing pans.

8. Cloth diapers

9. Desiccator containing drierite.

3.4.4.5 Procedure--

1. Assemble double-cylinder core sampler according to the instruction manual.

2. Prepare a flat soil surface at depth in profile to be sampled.

3. Drive core sampler into the soil with the driving head until the soil fills the brass or aluminum sleeve and extends slightly above it.

4. Remove driving head and twist double-cylinder core sampler.

5. Excavate soil on one side of the core sampler until the bottom of the cutting edge can be clearly seen.

6. To insure that the contact of the core with the main soil body is broken, run a knife across the bottom of the cutting edge. NOTE: Do this step taking care not to disrupt the soil core.

7. Pack a cloth diaper into the top of the double-cylinder core sampler until it rests on the top of the soil core and hold in place with one hand.

8. Gently tilt the top of the sampler towards the excavated side until the cutting edge of the sampler is exposed. Put the other hand across the bottom of the cutting edge to hold soil core in place. Remove core sampler from excavation.

9. Remove the core and sleeve from sampler by raising the cutting edge and applying gentle pressure to bottom of soil core while using the cloth diaper to insure that the soil core does not slide or fall from the sleeve.

10. Trim any excess soil off both ends of the soil core so a flat surface exists flush with the edges of the sleeve.

11. Remove the soil from the sleeve ring and place in a pint container lined with a plastic bag. Take care that no soil is lost in transfer.

12. Label the sample as to location, depth sampled and any other pertinent information.

13. Transfer the samples to the laboratory.

14. Weigh a labeled aluminum pan and record the weight (A).

15. Transfer the moist soil sample to the pan and record the weight (B).

16. Place the pan with sample in an oven and allow to dry for 24 hours at 105°C.

17. Remove the pan with sample from the oven and cool in a desiccator. Weigh pan and contents. Record weight (C).

3.4.4.6 Calculations--

1. Bulk Density = $(C - A)/347.5$ cc, where 347.5 cc is the volume of the cylinder.

2. Percent Field Moisture = $((B - C)/(C - A)) \times 100$.

3.4.5 Bulk Density (Saran Method)

3.4.5.1 Principle--

See 3.4.4.1

3.4.5.2 Comments--

Care should be exercised when handling methyl ethyl ketone. This chemical is toxic and flammable. An exhaust hood should be used during the mixing of the plastic solution. Containers used for storing the solvent and the plastic solution must have lids which provide a tight seal.

One sampling pit can be used to collect samples from several different depths. Start at the surface and work downwards. Take a sample at the surface and then remove all material until the horizontal layer at the desired depth is exposed. Then take sample and repeat process until all samples needed are collected.

When trimming a clod to the desired size, be careful not to compact or otherwise destroy it. Careful handling of the clod is necessary until final coatings of plastic have been applied.

3.4.5.3 Chemicals--

1. Water.

2. Methyl ethyl ketone ($CH_3COC_2H_5$).

3. Dow Saran F310 solution in methyl ethyl ketone. NOTE: This solution consists of 1 part Saran and 7 parts of methyl ethyl ketone. It is prepared as follows: Under an exhaust hood, add 2310 ml of methyl ethyl ketone to a 3.785 liter (1 gallon) paint can. Add 330 g of Dow Saran Resin F310 to the solvent. The plastic is mixed with an air-powered or nonsparking electric stirrer until the resin dissolves. If a high-speed stirrer is used, the resin should dissolve in about one hour. Seal the container tightly with lid to prevent evaporation of solvent. Care should be exercised when using methyl ethyl ketone since the solvent is flammable and its vapors mix with air to form explosive mixtures. Always work with this solvent under an exhaust hood.

3.4.5.4 Materials--

1. Tile spade and shovel.
2. Sharp knife.
3. Scissors.
4. Thread or fine wire.
5. Plastic bags (large enough to contain sample) with ties.
6. Boxes, heavy, cardboard (large enough to contain samples).
7. Cloth diapers or other suitable packing material.
8. Exhaust hood.
9. Beaker, 600 ml.
10. Balance, can be read to 0.1 g.
11. Weighing pan, aluminum or other metal.
12. Support stand with ring clamp.
13. Drying oven.
14. Wooden rolling pin.
15. Paper (to crush clods on).
16. Sieve with 2 mm openings (10 mesh).

3.4.5.5 Procedure--

1. Dig a pit from the surface of the soil downward until a vertical cross-section of the soil is exposed.

2. Starting at the surface, work downward and remove a section of soil larger than the clod to be studied from the face of the pit with a tile spade.

3. Take a soil clod, about 5 cm in diameter, weighing form 30 to 150 g from a larger piece of soil using a sharp knife to carefully cut away excess material.

4. Carefully break or cut off all protruding points, cut off all roots with scissors, and brush all loose materials from clod.

5. Loop thread or fine wire around clod and tie securely. Be sure to leave a loose end of at least 50 cm (20 in) of thread or fine wire.

6. Open can containing the plastic solution. Holding the clod by the loose thread or fine wire, immerse it in the plastic solution for 5-10 seconds.

7. Remove clod from plastic solution and suspend from a previously prepared line (like a clothes line) for 30 minutes to allow coating to dry. NOTE: Seal container containing plastic solution tightly to prevent evaporation of solvent.

8. When dry, place coated sample in airtight plastic bag. Label the sample. Record location, depth sampled, and other pertinent information in data book.

9. Put the bag in a rigid cardboard container to prevent breaking or crushing of clod. NOTE: To insure that sample bag will be immobilized, use cloth diapers for packing material around the plastic bag.

10. Transport sample to the laboratory.

11. Under an exhaust hood, open can containing plastic solution. Remove sample from plastic bag holding it by the loose thread or fine wire and immerse it in the plastic solution for 30 seconds.

12. Remove clod from plastic solution, reseal container of plastic solution, and hang clod on a line under the exhaust hood for 30 minutes.

13. Repeat steps 11 and 12 four more times.

14. Fill a 600 ml beaker with approximately 350 ml of water.

15. Place beaker, with water, on a balance and weigh it to the nearest 0.1 g. Record weight (A).

16. Attach a ring clamp to the top of a support stand and position stand so that the ring clamp extends over the beaker of water on the balance.

17. After the final coating has dried, take loose end of thread or fine wire and lower clod into beaker of water until clod is resting on the

bottom of the beaker. Record weight (B). NOTE: Do not allow loose end of thread to fall into the beaker.

18. Loop loose end of thread or fine wire over ring clamp and slowly raise clod off the bottom of beaker.

19. When clod is completely surrounded by water, record weight (C). NOTE: It is extremely important that the clod is not touching any part of the beaker and is entirely surrounded by water.

20. Remove clod from beaker and place on tray in an oven at 105°C for 48 hours.

21. Remove clod from oven, cool in a desiccator and weigh to the nearest 0.1 g. Record weight (D).

22. Take a knife and carefully cut plastic coating and thread or fine wire from clod.

23. Put all clod material on a sheet of paper and crush with a wooden rolling pin. NOTE: Be careful not to crush soft coarse fragments, but be sure to remove all fines from coarse fragments.

24. Pass crushed material through a 2 mm sieve.

25. Transfer all material caught on 2 mm sieve to a weighing pan and dry in an oven at 105°C for 4 hours.

26. Cool weighing pan and sample in a desiccator. Weigh to nearest 0.1 g and record weight (E).

27. Discard material and weigh weighing pan. Record weight (F).

3.4.5.6 Calculations--

1. Legend:

A = Weight of beaker and water.

B = Weight of beaker, water, and moist clod.

C = Weight of beaker, water, and moist clod suspended in water.

D = Weight of oven-dry clod.

E = Weight of weighing pan and clod material greater than 2 mm in effective diameter.

F = Weight of weighing pan empty.

V = Volume of moist clod.

X = Volume of coarse fragments in clod.

2. Bulk density of clod = D/V,

Where V = (C-A)/(1.00 g/ml), the density of water is assumed to be 1.00 g/ml.

3. Bulk density of the less than 2 mm material of the clod = [D - (E - F)]/(V - X), where: X = (E - F)/(2.65 g/ml) NOTE: This calculation assumes that all material greater than 2 mm in effective diameter has no porosity and has a particle density fo 2.65 g/ml.

4. Percent moisture of field sample on an oven-dry weight basis = [((B - A) - D)/D] X 100.

3.4.6 Bulk Density (Varsol Method)

3.4.6.1 Principle--

(See 3.4.4.1)

3.4.6.2 Comments--

The nonpolar liquid, Varsol, is used because of its availability, cheapness and absence of an offensive odor. Because of its nonpolar nature, it can replace air trapped in pores without causing the clod to slake like a polar liquid (water).

Clods used must hold together without breaking during routine field and laboratory work. When samples are packed for transportation to the laboratory, cushioning agents (i.e. diapers, styrofoam chips, crumpled paper) should be used to reduce the chances of clod breakage. Corrections can be made for soils containing coarse fragments using steps 28 through 31 in the procedure.

The density of each new container of Varsol should be determined by using a clean and dry 50 ml volumetric pipet and pipetting the Varsol into a clean and dry preweighed beaker. The weight of the Varsol is recorded to 0.01 g. The pipetting and weighing is repeated a total of three times. An average weight of the three readings is divided by 50 (ml of Varsol used to determine the density).

3.4.6.3 Chemicals--

Varsol - Trade name of EXXON cleaning fluid (but can usually be purchased from other suppliers). We have found Varsol to have a rather consistent density of 0.77 g/cc.

3.4.6.4 Materials--

1. Digging implements (spade and shovel).

2. Knife.

3. Plastic bags (large enough to contain sample) with ties.

4. Containers, rigid cardboard (large enough to contain samples).

5. Drying oven.

6. Thread (or similar light weight, thin cord).

7. Balance, can be read to 0.1 g.

8. Weighing pan (preferably aluminum, but glass or other metal can be substituted).

9. Blotting paper.

10. Desiccator apparatus, vacuum type, with hole in center of lid for a rubber stopper (Corning 3100 or equivalent). Supported above the bottom of the desiccator is a perforated porcelain desiccator plate having a large center hole. A two-hole rubber stopper is placed in the desiccator lid. In one hole is placed a 8 mm o.d., T-shaped tubing connector. From one end of the T-connector, a short piece of tubing with a hosecock clamp is applied to allow air back into the desiccator after evacuation. From the other end of the T-connector, attach a length of vacuum hose with a hosecock clamp to the vacuum source equiped with vacuum gauge. A short piece of 8 mm o.d. glass tubing (bent at 90°) is inserted into the second hole of the rubber stopper with the 90° bend being outside the desiccator. A length of tygon tubing is attached to the inside end of the glass tubingo so that when the desiccator is closed, the tubing extends below and through the center hole of the porcelain plate. Another piece of tygon tubing with hosecock is applied to the other end of the glass tubing and cut to extend to near the bottom of the Varsol container.

11. Support stand with ring clamp (aluminum rod can be substituted for the ring clamp).

12. Beaker, 600 ml.

13. Wooden rolling pin (optional).

14. Brown paper (optional).

3.4.6.5 Procedure--

1. Dig a pit from the surface of the soil downward until a vertical cross section of the soil is exposed through the depths to be sampled.

2. Remove a large layer of soil with a spade from the face of the sampling pit. Take a soil clod about 5 cm in diameter and weighing from 30 to 150 g from the layer of soil. NOTE: Use a knife to cut the clod from the soil. More than one clod can be taken for testing.

3. Put the sample in an airtight plastic bag. Label the sample. Record location, depth sampled, and other pertinent information in data book.

4. Put the bag in a rigid cardboard container to prevent breaking or crushing the clod. NOTE: To insure that sample bag will be immobilized, use cloth diapers for packing material around the plastic bag.

5. Transport the sample to the laboratory.

6. Weigh an oven-dry weighing pan and record the weight (A).

7. Carefully break off all protruding points and brush all loose material from the clod.

8. Loop thread around clod and tie leaving about 50 cm (20 in) of thread loose.

9. Place moist clod in weighing pan and weigh it to the nearest 0.1 g. Record weight (B).

10. Place moist clod on a small square of heavy blotting paper in the vacuum desiccator.

11. Apply grease to the ground glass surfaces of the lid and the bowl of the desiccator.

12. Place the lid on the bowl and make a tight seal. NOTE: Make sure the tubing extends below and through the center hole of the porcelain base plate in the bottom of the desiccator.

13. Clamp off the hoses that lead to the supply of Varsol and air inlet.

14. Evacuate to a pressure of less than 0.1 bar.

15. Clamp off hose leading to vacuum source.

16. Open clamp to hose leading to Varsol and admit fluid slowly until it completely covers sample.

17. After sample is completely covered with Varsol, allow sample to soak for one hour.

18. Fill a 600 ml beaker with enough Varsol to cover sample completely (approximately 350 ml) and weigh on balance to nearest 0.1 g. Record weight (C).

19. Take a support stand and attach a ring clamp at the top of stand. Position stand in such a manner that the ring clamp extends over the beaker of Varsol on the balance.

20. After soaking, open clamp on air inlet to allow the inside of the desiccator to return to atmospheric pressure.

21. Remove lid of desiccator carefully. Remove clod on its base of blotting paper from the fluid.

22. Separate blotting paper and clod. Carefully place clod into beaker of fluid on the balance pan and allow clod to rest on beaker bottom. Do not let the loose end of thread fall into the beaker. Record weight (D). NOTE: Separation of blotting paper and clod after removal of both from Varsol will eliminate a few drops of surplus fluid from the sample, but the drainage tension will be slight.

23. Take loose end of thread attached to clod and loop thread over the ring clamp (or straight rod) and slowly raise the clod off the bottom of beaker.

24. When clod is completely surrounded by fluid, record weight (E). NOTE: It is essential that clod is not touching the sides or bottom of the beaker and is entirely surrounded by the fluid.

25. Remove clod from beaker and place in weighing pan (pre-weighed in step 6). Allow samples to air dry overnight under a hood.

26. Dry clods in an oven at 105°C for 24 hours.

27. Remove samples from oven. Cool in desiccator and weigh to nearest 0.1 g. Record weight (F). NOTE: Steps 28 thru 31 are necessary if bulk density and porosity of the textural particles without coarse fragments are required.

28. Put clod on sheet of brown paper and crush with a wooden rolling pin. NOTE: Be careful not to crush small, soft coarse fragments, but be sure to remove all fines from coarse fragments.

29. Pass sample through a 2 mm sieve.

30. All material caught on 2 mm sieve is transferred to a weighing pan (pre-weighed in step 6) and dried in an oven at 105°C for 4 hours.

31. Cool weighing pan and sample in deiccator. Then weigh sample plus weighing pan and record weight (G).

3.4.6.6 Calculations—

1. Legend:

A = Oven-dry weight of weighing pan.

B = Weight of moist clod and weighing pan.

C = Weight of beaker and Varsol.

D = Weight of beaker, Varsol, and clod.

E = Weight of beaker, Varsol, and clod suspended in Varsol.

F = Oven-dry weight of clod and weighing pan.

G = Oven-dry weight of coarse fragments contained in clod and weighing pan.

X = Volume of water in clod (equals the volume of pore space filled with water).

Y = Volume of Varsol in clod (equals the volume of pore space filled with Varsol).

Z = Volume of clod.

T = Volume of coarse fragments.

Density of water = 1.00 g/cc.

Density of Varsol = 0.77 g/cc (see 3.4.6.2).

2. Bulk density of clod = (F - A)/Z, where:

Z = (E - C)/Density of Varsol.

3. Total pore space = X + Y, where:

X = (B - F)/Density of water; and

Y = [(D - C) - (B - A) - (F - A)]/Density of Varsol.

4. Total porosity = [(X + Y)/Z] X 100.

5. Bulk density of the less than 2 mm material in the clod.
Bulk density = (F - G)/(Z - T), where: T = (G - A)/2.65
NOTE: The coarse fragments are assumed to have no porosity; therefore, a particle density of 2.65 g/cc is used to find the volume of the coarse fragments. When coarse fragments have porosity the calculated bulk density and porosity of the fines (less than 2 mm material) will be incorrect, but bulk density and porosity of the whole clod, including coarse fragments, will be correct.

3.4.7 Bulk Density (Sand Method)

3.4.7.1 Principle--

See 3.4.4.1

3.4.7.2 Comments--

The calculated volume of the jar and attachment remain constant as long as

both maintain the same relative position to each other. If the two are to be separated, match marks should be made to permit reassembly to this position. The individual measured volumes of water (Q_1, Q_2, and Q_3) require filling the jar and attachment repeatedly (see 3.4.7.6, no. 1). Replicates should not differ more than 3 ml between the highest and lowest volume determined. Vibration of the sand during any of the weighings or density determinations may cause an increase in the sand bulk density and a decrease in accuracy. Sand bulk density (T) may change over time due to changes in moisture content or effective graduation. Field measurements should be run as soon as possible after the sand density (T) has been determined. Each new bag of sand must have its sand density determined (ASTM, 1974).

Care should be taken in excavating to minimize compaction of the soil surrounding the hole. Any material falling from the sides of the hole must be removed and placed with the material to be weighed. In this method, discrimination of very thin horizons is lost; however, due to the relatively large sample size, small errors in measuring the sand weight results in insignificant errors (Blake, 1965).

This method is especially suited to minesoils where coarse fragments prevent using a core sampler. The procedure also works well in coarse textured or unconsolidated materials that cannot be tested with either the Varsol or Saran techniques.

3.4.7.3 Chemicals--

Acetone (CH_3COCH_3) (optional).

3.4.7.4 Materials--

1. Template consisting of a thin, flat, metal plate 30.5 cm (12 in) square, with a 16.5 cm (6.5 in) diameter hole in its center.

2. Sand-funnel apparatus consisting of a lower cone flanged to 16.5 cm (6.5 in) to fit the above template and a top cone section that is threaded to receive the sand jug. A valve is located between the two cones to control the sand flow into the density hole (specifications in ASTM, 1974 p. 211).

3. A standard sand that is clean, dry, and free-flowing. Particle size should be uniform passing a sieve with 0.841 mm openings (20 mesh) and retained on a sieve with a 0.250 mm openings (60 mesh). (Ottawa sand or equivalent).

4. Balance, 20 kg (44.10 lb) capacity which can be read to 1.0 g (Model L-500 available from Soiltest, Inc., Evanston, IL or equivalent).

5. Large spoon.

6. Sand scoop.

7. Brown paper (optional).

8. Wooden rolling pin (optional).

9. Sieve, 2 mm openings (10 mesh).

3.4.7.5 Procedure (modified from ASTM, 1974)--

NOTE: Steps 1-16 and 3.4.7.6 no. 1-3 should be completed in the laboratory prior to going to the field. Steps 1-8 and 3.4.7.6 no. 1 must be completed when either the jar or funnel apparatus is replaced. Steps 9-12 and 3.4.7.6 no. 2 must be repeated for each new bag of sand. Steps 13-16 and 3.4.7.6 no. 3 must be repeated if the funnel apparatus is replaced.

1. Assemble apparatus and place match marks on both the jar and funnel apparatus to permit accurate realignment in case of separation.

2. Weigh assembled apparatus empty and record weight (A).

3. Place apparatus upright. Open valve and fill with water until the water appears over the valve.

4. Close valve and pour off excess water. Remove any water remaining in the funnel by sponging and then wiping dry.

5. Weigh apparatus filled with water. Record weight (B). Determine temperature of the water and record temperature (C).

6. Discard water in apparatus.

7. Repeat steps 3-6 two more times and determine the volume of the apparatus from an average of the three weighings.

8. Thoroughly dry apparatus by the addition of acetone to absorb water, followed by drying with a jet of moisture-free air or drying on a drying rack.

9. Place dry density apparatus upright on a firm, level surface. Close valve and fill funnel with sand.

10. Open valve and fill apparatus. NOTE: Keep funnel at least half full of sand during the filling procedures.

11. Close valve sharply and remove sand remaining in funnel.

12. Weigh apparatus filled with sand and record weight (D).

13. Invert apparatus and seat in template on a clean, level, planar surface.

14. Open valve and keep open until sand stops running.

15. Close valve sharply. Weigh apparatus and remaining sand. Record weight (E).

16. Replace sand following steps 9-11.

17. In the field, prepare the surface of the location to be tested so that it is a level plane.

18. Place template on surface.

19. Using a large spoon, dig the test hole inside the template hole, being careful to avoid disturbing the soil bounding the hole. NOTE: The excavated hole should have a diameter equal to the diamter of the template hole. The excavated walls of the finished hole should be as close to vertical as possible. The hole depth should be at least 7.6 cm (3 in) but not exceeding 16.5 cm (6.5 in) deep.

20. Place all loosened soil in a container, being careful not to lose any material.

21. Seat the density apparatus on the template and open the valve. After the sand has stopped flowing, close the valve sharply.

22. Weigh apparatus and remaining sand. Record weight (F).

23. Replace as much sand as possible from the hole back into the jar, being careful not to get contaminants in the sand from the hole.

24. Refill apparatus with sand using steps 9-11.

25. Preweigh a weighing pan and record weight (G).

26. Place moist material removed from the test hole on the preweighed pan. Record weight (H).

27. Place material in an oven at 105°C for 16 hours.

28. Cool in desiccator and reweigh. Record weight (I).
NOTE: Steps 29-32 are optional and are used when bulk density without coarse fragments is required.

29. Put excavated material on a sheet of brown paper and crush with a wooden rolling pin. NOTE: Be careful not to crush small soft course fragments, but be sure to remove all fines from coarse fragments.

30. Pass sample through a 2 mm sieve.

31. All material caught on a 2 mm sieve is transferred to weighing pan and dried in an oven at 105°C for 4 hours.

32. Cool weighing pan and sample in desiccator. Weigh sample plus weighing pan and record weight (J).

3.4.7.6 Calculations--

1. Legend:

A = Weight of empty apparatus.

B_1 = Weight of apparatus filled with water from first weighing (see 3.4.7.5, steps 2 through 7).

B_2 = Weight of apparatus filled with water from second weighing (see 3.4.7.5, steps 2 through 7).

B_3 = Weight of apparatus filled with water from third weighing (see 3.4.7.5, steps 2 through 7).

C = Temperature of water.

D = Weight of apparatus filled with sand.

E = Weight of apparatus and sand excluding sand in funnel.

F = Weight of apparatus and sand excluding sand in excavated hole and sand in funnel.

G = Weight of weighing pan.

H = Weight of moist sample and weighing pan.

I = Weight of oven-dry sample and weighing pan.

J = Weight of coarse fragments and weighing pan.

K = Volume of coarse fragments.

N_1 = Weight of water required to fill apparatus on first weighing (see 3.4.7.5, steps 2 through 7).

N_2 = Weight of water required to fill apparatus on second weighing (see 3.4.7.5, steps 2 through 7).

N_3 = Weight of water required to fill apparatus on third weighing (see 3.4.7.5, steps 2 through 7).

P_1 = Volume-temperature correction factor from Table 12 for first weighing (see 3.4.7.5, steps 2 through 7).

P_2 = Volume-temperature correction factor from Table 12 for second weighing (see 3.4.7.5, steps 2 through 7).

P_3 = Volume-temperature correction factor from Table 12 for third weighing (see 3.4.7.5, steps 2 through 7).

TABLE 12. VOLUME OF WATER PER GRAM BASED ON TEMPERATURE

Temperature (C) °C	Volume of Water (P) ml/g	Temperature (C) °C	Volume of Water (P) ml/g
12	1.00048	22	1.00221
14	1.00073	24	1.00268
16	1.00103	26	1.00320
18	1.00138	28	1.00375
20	1.00177	30	1.00435
		32	1.00497

Q_1 = Volume of water required to fill apparatus from first weighing (see 3.4.7.5, steps 2 through 7).

Q_2 = Volume of water required to fill apparatus from second weighing (see 3.4.7.5, steps 2 through 7).

Q_3 = Volume of water required to fill apparatus from third weighing (see 3.4.7.5, steps 2 through 7).

R = Average volume of density apparatus.

S = Weight of sand required to fill apparatus.

T = Bulk density of sand.

U = Weight of sand required to fill funnel.

V = Weight of sand required to fill excavated hole and funnel.

W = Volume of excavated hole.

Y = Weight of oven-dry sample

Z = Weight of moist sample.

2. $R = (Q_1 + Q_2 + Q_3)/3$, where:

$Q_1 = N_1 \times P_1$.

$Q_2 = N_2 \times P_2$.

$Q_3 = N_3 \times P_3$, and

$N_1 = B_1 - A$.

$N_2 = B_2 - A$.

$N_3 = B_3 - A$.

3. $T = S/R$, where:

$S = D - A$.

4. $U = D - E$.

5. $V = D - F$.

6. $W = (V - U)/T$.

7. Bulk density of soil = Y/W, where $Y = I - G$.

8. Percent moisture = $[(Z - Y)/Y] \times (100)$, where $Z = H - G$.

9. Bulk density of the less than 2 mm material in sample.
Bulk density = $[Y - (J - G)]/(W - K)$, where $K = (J - G)/2.65$.
NOTE: The coarse fragments are assumed to have no porosity; therefore, a particle density of 2.65 g/cc (density of quartz) is used to find the volume of the coarse fragments. See note under 3.4.6.6.

3.4.8 Particle Density

3.4.8.1 Principle--

The relationship of the solid soil particles to their total volume excluding the pore spaces between particles is called the particle density. It is normally expressed as grams per cubic centimeter. The mass of the solid particles is found by weighing and their total volume is determined by the displacement of a liquid whose mass and density are known (Blake, 1965).

3.4.8.2 Comments--

If measurements of volumes and weights are done carefully, this method is precise. A lack of precision in either measurement may result in serious error.

A non-polar liquid, Varsol, is used in the procedure instead of water because of the higher density values water gives for finely divided, active powders. Other polar liquids (e.g. toluene, xylene, or carbon tetrachloride) can be used, but they need special care in handling.

This measurement is used to mathematically determine porosity, airspace, and sedimentation rates for particle-size analysis. Minesoil samples are screened through a 2 mm sieve after rolling with a rolling pin. Sample is not ground with mortar and pestle.

3.4.8.3 Chemicals --

Varsol - Trade name of EXXON cleaning fluid (but can usually be purchased from other suppliers). We have found Varsol to have a rather consistent density of 0.77 g/cc.

3.4.8.4 Materials--

1. Pycnometer flask with ground glass lid (modified Hubbard-Carmick, Pyrex brand 1620 or equivalent).

2. Balance, can be read to 0.0001 g.

3. Vacuum desiccator.

3.4.8.5 Procedure (modified from Blake, 1965 and Gradwell, 1955)--

NOTE: All weights are recorded to ± 0.0001.

1. Oven dry the less than 2 mm sample at 60°C overnight.

2. Weigh a clean, dry pycnometer flask and lid. Record weight (W_a).

3. Add about 10 g of oven-dry sample to pycnometer. Clean outside and neck of pycnometer of any soil that may have spilled during transfer.

4. Weigh the pycnometer, including lid, and its contents. Record weight (W_s).

5. Fill pycnometer about one-half full with Varsol, washing any soil adhering to the neck into the pycnometer.

6. Place pycnometer into the vacuum desiccator, apply vacuum, and remove any entrapped air. Entrapped air will be removed when all bubbling ceases.

7. Remove the pycnometer and shake gently. NOTE: Repeat steps 6 and 7 until all bubbling ceases.

8. Fill the pycnometer with enough Varsol so that when the lid is put in place, the hole in the lid will be completely filled with Varsol.

9. Insert the lid and seat it carefully.

10. Thoroughly dry and clean the outside of the pycnometer with a dry cloth.

11. Weigh the pycnometer and its contents. Record weight (W_{sv}).

12. Remove sample and Varsol from the pycnometer. NOTE: Thoroughly wash pycnometer and lid with Varsol to insure removal of sample.

13. Fill pycnometer with enough Varsol so that the hole in the lid will be filled with Varsol when the lid is seated.

14. Insert and seat lid. Thoroughly dry the outside with a dry cloth.

15. Weigh pycnometer filled with Varsol. Record weight (W_v).

3.4.8.6 Calculations--

Particle density (Dp) = $dv\ (W_s - W_a)/[(W_s - W_a) - (W_{sv} - W_v)]$, where:

dv = Density of Varsol in g/cc (see note below).

W_s = Weight pycnometer plus sample.

W_a = Weight of pycnometer filled with air.

W_{sv} = Weight of pycnometer filled with sample and Varsol.

W_v = Weight of pycnometer filled with Varsol.

NOTE: The density of Varsol must be determined for each new supply of Varsol. Using a pipette, add exactly 50 cc to a previously tared beaker. Record weight of the Varsol.

dv = weight (g) of Varsol/50 cc.

3.4.9 Total Porosity

3.4.9.1 Principle--

The bulk volume of a field moist soil sample contains soil particles, moisture, and air. The portion of the bulk volume filled with moisture and air is called pore space. Bulk density measurements (3.4.4-3.4.7) are calculated by dividing the oven-dry weight of the mass (in grams) by the bulk volume. This value is considerably lower than the average particle density (3.4.8). This means that part of the bulk volume is pores filled with air. Calculation of total porosity is done by converting data from densities into volumes. The volume (VB) of the bulk sample is derived from a bulk density measurement. The volume (VP) is the collective volume occupied by solid particles and is derived from the particle density measurement. Therefore, VP/VB is the fraction of the volume occupied by solid particles. In this manner, total porosity can be calculated using the equation in 3.4.9.6 (Vomocil, 1965).

3.4.9.2 Comments--

Total porosity can be measured directly if the "Varsol" method is used to find the bulk density. The procedure and calculations are given in 3.4.6.

Do not use an assumed particle density of 2.65 g/cm^3 if carbolithic materials are present in the sample in appreciable amounts. Measure the particle density of this material using 3.4.8.

3.4.9.3 Chemicals--

None required.

3.4.9.4 Materials--

None required.

3.4.9.5 Procedure--

1. Determine the bulk density using one of the following methods: (a) 3.4.4; (b) 3.4.5; (c) 3.4.6; or (d) 3.4.7.

2. Determine particle density using method 3.4.8. NOTE: In cases where great accuracy is not required, use the assumed value of 2.65 g/cm^3 for the particle density of mineral soils.

3.4.9.6 Calculations--

1. TP = Total porosity: percentage of the bulk volume not occupied by solids.

2. BD = Bulk density of soil.

3. PD = Particle density of soil.

4. $TP = [(PD - BD)/PD] \times 100$.

3.4.10 Free Swelling (Settling Volume)

3.4.10.1 Principle--

Swelling is an innate property of the clays. Swelling may arise in two different ways: (1) water molecules becoming positioned between the particles of clay; (2) water molecules becoming positioned within the molecular structure of the clay mineral. Kaolinite and mica-like clays will only exhibit swelling due to the former process; therefore, these clays will have limited volume change, especially kaolinite. Clays of the montmorillonite type exhibit extensive swelling mainly because of the latter process. Free swelling is an important property of this type of clay mineral (Marshall, 1949).

3.4.10.2 Comments--

Step number 4 of 3.4.10.5 (procedure) should be performed very carefully and slowly so that no sample is lost. Also, step number 10 should be performed exactly as described.

This simple method can be used effectively to evaluate the stability of materials. Materials exhibiting extensive swelling would be unstable on steep slope, haul-road, etc. Also, future land use would be affected by such materials.

3.4.10.3 Chemicals--

Distilled water (H_2O).

3.4.10.4 Materials--

1. Graduated cylinders, 100 ml capacity with 1 ml graduations.
2. Powder funnels.
3. Sieve, 0.25 mm openings (60 mesh).
4. Polypropylene wash bottle.
5. Balance, can be read to 0.001 g.
6. Pencil, yellow or any color that can be seen easily through turbid water.
7. Standard liquid limit device (Sowers, 1965, Fig. 1-1, p. 395) adjusted to drop a distance of 1 cm.

3.4.10.5 Procedure--

1. Weigh a 10.00 g air-dry sample of earthy material ground to pass a 60 mesh sieve.
2. Fill a 100 ml graduated cylinder to the 85 ml mark with distilled water.
3. Put a powder funnel in the neck of the graduated cylinder.
4. Slowly add the 10.00 g of earthy material to the graduated cylinder in several small increments. NOTE: This step must be done slowly so that all earthy material is transferred into the cylinder without unnecessary entrapment of air.
5. Add distilled water to the cylinder until the liquid level reaches the 100 ml mark, washing off any particles adhering to the sides of the cylinder.
6. Set cylinder aside and let stand undisturbed for 6 hours.
7. At the end of 6 hours, place cylinder on cup of liquid limit device and turn crank 30 times at a rate of one revolution per second. NOTE: After every five revolutions straighten cylinder without changing the rate if necessary to keep cylinder upright.
8. Set cylinder aside and let stand undisturbed for an additional 18 hours.

9. At the end of the prescribed time, take a yellow pencil and place it behind the cylinder so that the pencil can only be seen by looking through the cylinder and the material in the cylinder.

10. Starting at the top of the cylinder, lower pencil down the back of the cylinder until the pencil can no longer be seen.

11. Record the volume at the point where the pencil cannot be seen.

3.4.10.6 Calculations--

Free swelling (settling volume) is expressed on a volume per mass basis (cc/g).

Free swelling = volume/10.00 g air-dry sample.

3.4.11 Moisture Retention (Pressure Plate Method)

3.4.11.1 Principle--

The amount of work needed to remove water from soil is measured by the pressure plate apparatus. This work equals the energy with which the soil sample holds the water. In this procedure a saturated soil sample rests on a semipermeable membrane and is subjected to controlled pressures in excess of atmospheric pressure. A water continuum, which is at atmospheric pressure outside the apparatus, exists from the surface of the soil sample to the open-air side of the semipermeable membrane; therefore, the compressed gas forces water out of the pores of the sample through the membrane by way of the water continuum. Water outflow from the chamber ceases when equilibrium has been reached (i.e., when the pressure exerted by the gas is counteracted by the tension (negative pressure) with which the soil particles hold onto the water). It is possible to determine directly the moisture content of the soil at that particular tension. Normally a curve called the moisture characteristic curve is developed by equilibrating soils at pressures from 0 through 15 bars or higher (Richards, 1965).

3.4.11.2 Comments--

Errors in these measurements can come from many sources. Some of the principle errors come from nonrepresentative subsamples, losses due to evaporation during the approach to equilibrium due to a leak in the air pressure chamber of the semipermeable membrane, pressure to temperature effects in excess of 1°C causing hysteresis effect, failure to obtain outflow equilibrium, and inadequate pre-wetting of samples. Additional errors can also come from evaporation losses when the samples are being removed from the chamber and loss of sample during removal from the chamber; however, these errors can be overcome as the operator becomes more proficient.

The semipermeable membrane, which may be a ceramic plate or cellulose disc, has a definite bubbling pressure. Below bubbling pressure of these membranes, the membrane will allow free movement of moisture from one side to the other; however, soil particles and air are not transmitted. The membrane contains pores which are full of water and form the continuum from the soil sample through the membrane to the atmosphere on the outside. When the bubbling pressure of a membrane has been exceeded, some pores contain gas instead of water and the gas moves freely through the membrane and pressure is lost. After the system and the apparatus have been checked, determine which pressure range will be measured. The USDA - SCS commonly measures moisture retention at 1/3 and 15 bar tensions; however, the range of tensions of prime interest for plant growth may be from 0 to 2 bars or other ranges. For highly disturbed soils, coarse fragments sometimes constitute a major part of the soil volume. Therefore, the particle sizes used to get a moisture characteristic curve are not necessarily the same as for soils with few coarse fragments. Soils sieved to contain only particle sizes of less than 6.35 mm effective diameter are used at West Virginia University to determine a moisture characteristic curve. Also, moisture characteristic curves can be determined for the particle size range of 6.35 mm to 2 mm in effective diameter, as well as for the less than 2 mm particles (Richards, 1965).

3.4.11.3 Chemicals--

1. Distilled water.

2. Compressed nitrogen gas.

3.4.11.4 Materials--

1. Five bar pressure plate extractor (Soil Moisture Equipment Company Catalog No. 1600 or equivalent).

2. Pressure control manifold, accuracy of control within 1/100 psi in the 0.50 psi range (Soil Moisture Equipment Company Catalog No. 700-3 or equivalent).

3. One bar pressure plate cells (Soil Moisture Equipment Company Catalog No. 1290 or equivalent).

4. Three bar pressure plate cells (Soil Moisture Equipment Company Catalog No. 1690 or equivalent).

5. Soil sample retaining rings (Soil Moisture Equipment Company Catalog No. 1093 or equivalent).

6. Connecting hose (Soil Moisture Equipment Company Catalog No. 1293 or equivalent).

7. Nitrogen gas tank gauges - 1 for tank pressure and 1 for outflow pressure.

8. Large spatula or small pancake turner.

9. Wax paper.

10. Plastic teaspoon.

11. Balance, can be read to 0.01 g.

12. Drying oven.

13. Aluminum pans, for weighing samples.

14. Laboratory notebook.

15. Desiccator, with silica gel desiccant.

3.4.11.5 Procedure--

NOTE: This apparatus and procedure are used for negative pressures of 0 to -3 bar. Read instrument's instruction manual before starting procedure.

1. Check pressure in the nitrogen tank.

2. Check all fittings by pressurizing system. NOTE: Take a toothbrush and a bar of soap and mix up a soapy foam. Brush foam over each fitting to see if there are any leaks in the system when pressurized.

3. Check ceramic plates by forcing compressed air into outlet valve. Seal off valve and submerge ceramic plate in pan of water. If any bubbles appear, there is a hole in the rubber gasket sealed to the plate. Repair the leak or do not use the plate.

4. Place the ceramic plate to be used in a pan of distilled water and soak overnight (12-16 hrs). This is done when the ceramic plates have been dried over a period of time. If the ceramic plate has been used for a previous determination, this prolonged soaking is not necessary.

5. Take the aluminum pans and place a soil sample retaining ring inside the pan. Draw a line around the top of the ring so that the approximate height of the ring is outlined on the inside of the aluminum pan. The desired volume of subsample that would be put into the aluminum pan would be slightly less than needed to fill the soil sample retaining ring.

6. Use a thin plastic teaspoon and lift the soil from the container and fill the aluminum pan to the volume mark. Do two replicates in the same manner. NOTE: Be sure that all the pans are marked with the soil sample number.

7. After the ceramic plate has been soaked overnight, place the soil sample retaining rings on the ceramic plate in such a fashion that a diagram can be easily made of the set up showing the sample number for each particular ring.

8. Take the aluminum pan containing the approximate volume of soil sample needed and carefully dump it into the proper soil sampling retaining ring on the ceramic plate. Take the spatula or the spoon and carefully flatten

the sample until it is level with the top edge of the soil sample retaining ring. NOTE: Do not compact this material. Just carefully flatten by spreading.

9. After all the soil samples have been placed on the soaked ceramic plates, add an excess of water to the surface of the ceramic plate and allow the samples to soak for 16 hours. NOTE: Be sure there is enough water on the ceramic plate to allow samples to wet without removing water from the pores of the plates.

10. Cover samples and ceramic plate with wax paper to prevent evaporation.

11. After the samples have soaked overnight (16 hours), remove the excess water from the surface of the ceramic plate by means of a pipette.

12. Remove the wax paper from the soil samples. Connect the outflow tube on the ceramic plate to the outflow tube on the wall of the extractor.

13. Cover the extractor with the metal top. NOTE: Be sure that the "O" ring seal is in place.

14. Clamp the lid to the bottom of the extractor with clamping bolts. Tighten the wing nuts on the clamping bolts by hand.

15. With the needle valve, the "Nullmatic" type regulator, and the coarse adjustment regulator on the manifold all closed, pressurize the system by means of the controls on the nitrogen tank. Turn the "Nullmatic" type regulator valve to wide open and use the coarse adjustment valve on the manifold to get a reading on the pressure gauge of very slightly in excess of the desired pressure.

16. Use the "Nullmatic" type regulator to get the desired pressure reading on the manifold's pressure gauge.

17. Slowly open the needle valve at the end of the manifold and pressurize the pressure plate extractor. NOTE: Two hours after system is pressurized, check pressure gauge on manifold for any final adjustment.

18. Samples that are 1 cm high can be removed any time after 48 hours from initiation of the extraction. Some soils approach equilibrium in 18 to 20 hours; therefore, after 20 hours the outflow tube is tested periodically with blotter paper. If no moisture accumulates on the blotter paper after it has been held against the outflow tube for approximately 1 minute, equilibrium has been reached and the extraction can be stopped.

19. Clean aluminum pan previously used. Oven dry, cool in desiccator, and weigh to nearest 0.01 g. Record weight (A).

20. Put a piece of tubing over the outflow tube and clamp the tubing off with a pinch clamp. Shut the pressure source off, then drain the system of compressed gas slowly by using the coarse adjustment valve on the manifold.

21. After the system has been drained of compressed gas, disconnect the hose leading to the extractor. NOTE: This will insure that the extractor is no longer pressurized.

22. Remove the clamping bolts and extractor lid.

23. Remove the samples one at a time and place in weighed aluminum pans.

24. Quickly weigh the aluminum weighing pan and the sample. Record weight (B).

25. Place samples in the drying oven at 105°C. Allow samples to dry overnight.

26. Remove samples from drying oven and place in a desiccator filled with silica gel desiccant. Allow samples to cool.

27. Weigh samples and weighing pan. Record weight (C).

28. Discard sample.

29. Make sure that the pressure at which the extraction was carried out is recorded in the laboratory notebook.

3.4.11.6 Calculations--

1. Legend:

A = Weight of aluminum weighing pan.

B = Weight of moist sample and aluminum weighing pan.

C = Weight of aluminum weighing pan and oven-dry sample.

2. Percent moisture = $[(B - C)/(C - A)] \times 100$.

3.4.12 Moisture Retention (Pressure Membrane Method)

3.4.12.1 Principle--

See 3.4.11.1

3.4.12.2 Comments--

See 3.4.11.2

3.4.12.3 Chemicals--

1. Distilled water.

2. Compressed nitrogen gas.

3.4.12.4 Materials--

1. Pressure membrane extractor (Soil Moisture Equipment Company Catalog No. 1000 or equivalent).

2. Pressure control manifold, 0-225 psi range with Mercury Differential Regulator (Soil Moisture Equipment Company Catalog No. 700-1 or equivalent).

3. Torque wrench and socket (Soil Moisture Equipment Company Catalog No. 1090 or equivalent).

4. Two connecting hoses (Soil Moisture Equipment Company Catalog No. 1091 or equivalent).

5. Soil sample retaining rings (Soil Moisture Equipment Company Catalog No. 1093 or equivalent).

6. Cut cellulose membrane discs (Soil Moisture Equipment Company Catalog No. 1096 or equivalent.

7. Two nitrogen gas tank gauges - one for tank pressure (0-4,000 psi) and one for outflow pressure (0-500 psi).

8. Large spatula or small pancake turner.

9. Wax paper.

10. Plastic teaspoon.

11. Balance, can be read to 0.01 g.

12. Drying oven.

13. Aluminum pans, for weighing samples.

14. Laboratory notebook.

15. Desiccator, with silica gel desiccant.

3.4.12.5 Procedure--

NOTE: Read instrument's instruction manual before starting procedure.

1. Place a cut cellulose membrane disc in a pan of distilled water and allow disc to soak for at least 30 minutes.

2. Check pressure in the nitrogen tank.

3. Check all fittings by pressurizing system. NOTE: Take a toothbrush and a bar of soap and mix up a soapy foam. Brush foam over each fitting to see if there are any leaks in the system when pressurized.

4. Take the aluminum pans and place a soil sample retaining ring inside the pan. Draw a line around the top of the ring so that the approximate height of the ring is outlined on the inside of the aluminum pan. The desired volume of subsample that would be put into the aluminum pan would be slightly less than needed to fill the soil sample retaining ring.

5. Use a thin plastic teaspoon and lift the soil from the container and fill the aluminum pan to the volume mark. Do two replicates in the same manner. NOTE: Be sure that all the pans are marked with the soil sample number.

6. Remove the screen drain plate from the base of the pressure membrane extractor. Clean the screen to remove all soil grains that might puncture the membrane. Wet screen drain plate with distilled water and be sure that the drain hole is open.

7. Place the screen drain plate in its proper position. Remove the cellulose membrane disc from the water and place it on the screen drain plate. NOTE: The membrane should completely cover the screen drain plate. Arrange the membrane so there is a minimum of wrinkling. The membrane cannot be handled in this manner when it is dry because cracking will occur.

8. Place an "O" RING on the cellulose membrane. Put the standard cylinder (16 mm high) on top of the "O" RING. NOTE: Be sure that the "O" RING is in the lower groove of the standard cylinder and that the air-entry is pointing to the back of the pressure membrane extractor where the PM Hinge is mounted.

9. Latch the turn buttons (eccentric clamping screw assembly) into the grooves on the outside of the standard cylinder and tighten wing nuts. NOTE: The turn buttons hold everything in place when the samples are left to soak overnight.

10. Place soil sample retaining rings on the membrane. Draw a diagram of the arrangement of the rings on the membrane using the air-entry port as a guide. NOTE: A sample number is shown on the diagram for each soil sample retaining ring.

11. Attach a short piece of rubber tubing to the outflow tube on the bottom of the screen drain plate. Close off the outflow tube by attaching a pinch clamp to the rubber tubing.

12. Take the aluminum pan containing the approximate volume of soil sample needed and carefully dump it into the proper soil sampling retaining ring on the membrane. Take the spatula or the spoon and carefully flatten the sample until it is level with the top edge of the soil sample retaining ring. NOTE: Do not compact this material. Just carefully flatten by spreading.

13. Add an excess of water to the surface of the membrane and allow the samples to soak for 16 hours. NOTE: Be sure there is enough water on the membrane to allow samples to wet without removing water from the pores of the membrane.

14. Cover samples and membrane with wax paper to prevent evaporation.

15. After the samples have soaked overnight (16 hours) remove the excess water from the surface of the membrane by means of a pipette.

16. Place an "O" RING in the groove on the top of the standard cylinder.

17. Depress the PM hinge, put the lid in place and close the cell. NOTE: Be sure that the rubber diaphragm is between the lid and the top "O" RING before closing the cell.

18. Bolt the pressure membrane extractor shut using a torque wrench to tighten the bolts uniformly. A torque of 25 foot-pounds is usually adequate for air pressure up to 15.5 bars (225 psi).

19. The connecting hose coming from the mercury differential regulator is attached to the air-entry port on the side of the standard cylinder.

20. The other connecting hose is attached to the air-entry port on the top of the lid.

21. Remove pinch clamp from rubber tubing on outflow tube on bottom of pressure membrane extractor. Put 100 ml beaker under outflow tube and catch excess water.

22. Pressurize the system up to the first regulator on the manifold by turning the tank regulator on. Set the gas pressure in the line 2 bars (29 psi) higher than the desired cell pressure.

23. Open the bypass valve on the mercury differential regulator.

24. Admit gas into the cell slowly using the regulator on the manifold until the desired pressure is attained.

25. After about 2 hours, or when the outflow rate has decreased appreciably, close the bypass valve at the top of the "U" tube and open the exhaust valve on the air pressure test gauge side of the manifold. When gas is heard bubbling past the mercury in the "U" tube, close the exhaust valve and readjust the gas pressure using the first regulator on the manifold. NOTE: The membrane should be tested for leaks by submerging the rubber tubing connected to the outflow tube in a beaker of water. If there is rapid bubbling and/or a hissing of gas can be heard, then there is a leak in the membrane. The gas should be shut off and the procedure started again using a new membrane.

26. Check the pressure gauge reading after a few hours and readjust the gas pressure if needed.

27. Samples that are 1 cm high can be removed any time after 48 hours from initiation of the extraction. Some soils approach equilibrium in 18 to 20 hours; therefore, after 20 hours the outflow tube is tested periodically with blotter paper. If no moisture accumulates on the blotter paper after

it has been held against the outflow tube for approximately 1 minute, equilibrium has been reached and the extraction can be stopped.

28. Clean aluminum pan previously used. Oven dry, cool in desiccator, and weigh to nearest 0.01 g. Record weight (A).

29. Attach piece of tubing to the outflow tube and clamp with a pinch clamp. Open the bypass valve and shut the pressure source off. Drain the system of compressed gas slowly using the first regulator on the manifold.

30. After the system has been drained of compressed gas, disconnect the hoses leading to the top and side of the extractor. NOTE: This will insure that the extractor is no longer pressurized.

31. Remove the clamping bolts, extractor lid, and rubber diaphragm.

32. Remove the samples one at a time and place in weighed aluminum pans.

33. Quickly weigh the aluminum weighing pan and the sample. Record weight (B).

34. Place samples in the drying oven at 105°C. Allow samples to dry overnight.

35. Remove samples from drying oven and place in a desiccator filled with silica gel desiccant. Allow samples to cool.

36. Weigh samples and weighing pan. Record weight (C).

37. Discard sample.

38. Make sure that the pressure at which the extraction was carried out is recorded in your laboratory notebook.

3.4.12.6 Calculations—

1. Legend:

A = Weight of aluminum weighing pan.

B = Weight of moist sample and aluminum weighing pan.

C = Weight of aluminum weighing pan and oven-dry sample.

2. Percent moisture = $[(B - C)/(C - A)] \times 100$.

3.5 MICROBIOLOGICAL METHODS

3.5.1 Summary

Early soil microbiologists developed and published original versions of the procedures described in this publication. These procedures were used in soil investigations by their contemporaries and later soil microbiologists the world over. The data obtained have played an important role in our continual quest to unravel the mysteries of the soil.

These procedures, on the whole, are simple, easy to use, and require a minimum of equipment. Because they were developed years ago, none of the so-called "modern sophisticated" laboratory apparatus is involved.

These procedures were used in minesoil or strip mine spoil investigations over a 15-year period. Some were chosen because no better method was available, or because of a lack of equipment. These generally simple methods used in studying minesoils have again played an important role as they did earlier on conventional soils.

Though mainly simple procedures, careful planning, careful work, and a conscientious worker are basic requirements. A technician can be trained to perform the routine laboratory work described. Complex biological interpretations of these laboratory measurements in relation to field problems should include a person knowledgeable in soil microbiology.

3.5.2 Buried Slide Technique

3.5.2.1 Principle--

This technique was developed independently by both Cholodony (1930) and Rossi et al. (1936). It is a simple procedure and provides useful information concerning the microbes, particularly to their spatial relationships to each other, plant roots, debris, and soil particles. If the organisms remain intact, observations may be made of colony characteristics, feeding of organisms on materials, and response of organisms to environmental factors, such as water films (Frederick, 1965).

3.5.2.2 Comments--

The method is not quantitative but can be used to show microbial differences among various treatments of a native soil or minesoil. Burying two or more slides in each minesoil and/or treatment and removing one from each at weekly intervals will yield information on relative abundance and associations of the microbes.

Often the actual microorganism is no longer attached to the slide, but after staining, the size, shape, and location of the missing entity is revealed by stain deposition. This often reveals locations where organic debris and sometimes soil aggregates have been in contact with the slide.

Some determination of individual organisms can be made by placing the slide flat on the surface of an agar medium plate. The plate is incubated 2-3 hours, the slide aseptically removed, then incubation continued for at least 24 hours. This procedure will require duplicate slides, as a slide used in this manner is no longer useful for staining and microscopic observation and most organisms on a stained slide are dead.

One familiar with bacteria, fungi, actinomyces and diatoms, for example, will find the interpretation of the microscopic examination of a stained contact slide much easier than one without such familiarity. The technique is more useful when used in conjunction with other microbial methods for soil microbial studies than when used alone.

3.5.2.3 Chemicals--

NOTE: All chemicals must be ACS Certified pure grade.

1. Phenol (C_6H_5OH), 5% aqueous: Dissolve 5.0 g of phenol in distilled water and dilute to a volume of 100 ml.

2. Phenolic rose bengal stain: Weigh 1.0 g of rose bengal. Add 100.0 ml of 5% aqueous phenol. Add 0.05 g certified grade calcium chloride ($CaCl_2$).

3.5.2.4 Materials--

1. Straw, autoclave-sterilized, ground to 40 mesh, or any other material under study.

2. Alfalfa, autoclave-sterilized, ground to 40 mesh, or any other material under study.

3. Samples from the top 13 cm (5 in) of a soil (or any constant depth under study).

4. Sieve, 2 mm openings (10 mesh).

5. Sterile straight-sided water tumblers, or similar glass containers such as beakers.

6. 7.62 X 2.54 cm (3 X 1 in) sterile glass microscope slides.

7. Microscope with 10X or 15X eyepiece and 97X oil immersion objective.

8. Autoclave, steam, capable of holding 15 psi and 121°C.

9. Hilgard soil cups. NOTE: A sieve with 1 mm openings can be used.

10. Spatula.

11. Filter paper

12. Humid chamber. NOTE: A container large enough for the pan that will retain moisture can be used.

13. Balance, can be read to 0.01 g.

14. Drying oven.

15. Glass rods.

16. Pan. NOTE: Any pan that will hold the cups can be used.

3.5.2.5 Procedure (modified from Allen, 1949; Wilson and Hedrick, 1957a; Frederick, 1965)--

1. Place a circle of filter paper, cut to fit exactly, on the brass perforated bottom of a Hilgard cup, and moisten the filter paper.

2. Weigh the complete unit. Record weight (A).

3. Fill the cup with air-dried minesoil.

4. Compact the minesoil by dropping the cup 10 times through a distance of approximately 3 cm (1 in).

5. Level the soil surface with a spatula.

6. Weigh cup and minesoil. Record weight (B).

7. Lay two glass rods on the bottom of the pan.

8. Place the cup of minesoil on glass rods.

9. Add water to the pan to reach about half cup height.

10. Allow the soil to become saturated and remain in pan and water for 24 hours.

11. Remove cup, carefully wipe outside cup surfaces and underneath bottom to remove adhering water.

12. Weigh the cup with the soil in a saturated condition. Record weight (C).

13. Place cup in drying oven for 24 hours at 105°C.

14. Cool in desiccator and weigh immediately. Record weight (D). Remove soil, brush cup and filter paper free of soil and weigh immediately. Record weight (E).

15. Calculate soil moisture of the air-dried soil as well as the water holding capacity (see 3.5.2.6).

16. Prepare glass microscope slides by cleaning them thoroughly. If desired, flame slides just before use to insure sterility. NOTE: It is desirable to use new slides.

17. Pass samples through 2 mm hardware cloth sieve to remove the rocks and pieces of coal.

18. To 3 tumblers containing 150 g (oven-dried at 105°C for 16 hours)or some constant weight of soil, add the following: (First tumbler) no treatment -

control; (Second tumbler) soil thoroughly mixed with 0.5% autoclave-sterilized ground straw; (Third tumbler) soil thoroughly mixed with 0.5% autoclave-sterilized ground alfalfa.

19. Bring the soil moisture to 50% of the sample's water-holding capacity in the glass containers. Add slightly more so the treatment material will be moistened without diminishing the 50% water-holding capacity.

20. Insert carefully the prepared glass slides (2 per tumbler) vertically into the soil leaving about 13 mm (0.5 in) of each slide above the surface.

21. Press soil gently against slide.

22. Weigh tumbler, sample, and slides.

23. Cover tumblers with paper caps to prevent excessive evaporation, but not to exclude aeration.

24. Incubate soil tumblers at room temperature for one week.

25. Add water during incubation (about twice a week) to replace that lost by evaporation. NOTE: Add water until weight of sample and tumbler is same as weight found in step 22.

26. After incubation remove soil from only one side of one slide using a spatula. NOTE: Gently break the slide away from the soil without sliding the slide.

27. Remove large clumps of sand and soil from the slide surface to be observed by means of a dissecting needle or some small sharp pointed instrument.

28. Air dry slide.

29. With the aid of a small gentle stream of water, remove excess soil from the undisturbed side until only a thin film remains.

30. Clean disturbed side with a damp cloth. NOTE: This is the side that will not be stained.

31. Air dry slide.

32. Fix slide by passing it over a bunsen burner at low flame four or five times. Do not cook. This "fixes" the material on the slide reducing the likelihood of loss during the staining procedure.

33. Place the fixed slide over a steam bath (or beaker of boiling water). Flood slide for 6 to 10 minutes with phenolic rose bengal. NOTE: Avoid drying slide by adding stain as needed.

34. Remove excess stain by washing the slide gently with water until no more stain is removed.

35. Air dry slide.

36. Examine the slide microscopically using a 10X or 15X eyepiece with a 97X oil immersion objective. CAUTION: Avoid scratching oil immersion objective by making sure that all soil particles have been removed from the slide.

37. Continue incubation of second slide for another week. Examine slide in identical manner.

38. Examine at least 5 fields per slide.

39. Make drawings of representative fields. Arrange the drawings in two rows so that a comparison of the slides per treatment can be readily observed.

3.5.2.6 Calculations--

1. Legend:

A = Weight of cup and moist paper.

B = Weight of cup, moist paper, and air-dried soil.

C = Weight of cup, moist paper, and saturated soil.

D = Oven-dry weight of cup, paper, and soil.

E = Oven-dry weight of cup and paper.

S = Weight air-dry soil.

T = Weight saturated soil.

U = Weight oven-dry soil.

V = Weight water in air-dry soil.

W = Weight water in saturated soil (water loss).

X = Percent moisture in air-dry soil.

Y = Percent water-holding capacity of soil.

Z = Grams of water per 100 g oven-dry soil needed to make 50% water-holding capacity.

2. $S = B - A$.

3. $T = C - A$.

4. $U = D - E$.

5. V = S - U.

6. W = T - U.

7. X = (V/U) X 100.

8. Y = (W/U) X 100.

9. Z = Y/2.

3.5.3 Total Microbial Count (Agar-Plate Method)

3.5.3.1 Principle--

When a soil dilution is dispersed in appropriate agar medium and incubated under favorable conditions, discrete and macroscopically visible colonies of microorganisms will develop. Calculations using the number of colonies developing on the agar will give the "total count." The total count obtained, however, is only a fraction of the total number of microbes present. If the conditions are uniform throughout, relative if not absolute, microbial populations can be counted successfully (Clark, 1965).

3.5.3.2 Comments--

The agar-plate method is highly empirical. Care must be taken of details in the technique if individual workers are to obtain comparable results.

Soil samples should be processed the same day they are collected in their natural, undried condition. Drying of the soil reduces the total count, whereas storing moist samples at room temperature more than one day increases the total count.

Primary soil sample dilutions should be withdrawn within ten minutes after shaking. Rapid multiplication of organisms may result if counting is delayed. All samples should be withdrawn from the middle of the suspension immediately after vigorous hand shaking, since soil particle settling tends to move microorganisms to the bottom of the suspension. Care should be taken not to count soil particles that have settled from the solution as colonies.

The melted medium must be cooled to a temperature of 42° to 45°C before mixing with the soil, as some of the organisms are killed at higher temperatures. If the flask containing the melted medium is too hot when touched to the cheek, it's too hot for microorganisms.

Although the procedure uses soil-extract agar, egg-albumen, or yeast-extract agar can be used (Clark, 1965).

3.5.3.3 Chemicals--

NOTE: All chemicals must be ACS Certified pure grade.

1. Soil-extract (Lockhead, 1940): Mix the following: 1000.0 g of fertile soil and 1500.0 ml of distilled water. Autoclave mixture for 30 minutes at 15 psi. After partial cooling and settling, filter suspension using a Buchner funnel, filter-aid, and medium-grade filter paper. If extract cannot be filtered easily in this way, pour turbid soil:water suspension into a 2 liter graduated cylinder and let stand in a refrigerator at 4°C overnight. Settling and clearing will usually result.

2. Soil-extract agar (Lockhead, 1940): Mix 20.0 g agar, 0.5 g dipotassium phosphate (K_2HPO_4), and 0.1 g of dextrose with 1000.0 ml of soil-extract. Adjust pH to between 6.8 and 7.0 with 3 *N* HCl or 3 *N* NaOH. Sterilize medium by autoclaving at 15 psi for 15 minutes.

3. Egg-albumen agar (Waksman and Fred, 1922): Dissolve 0.25 g of egg albumen in 10 ml of 0.1 *N* NaOH. Add 15.0 g agar, 1.0 g dextrose, 0.5 g dipotassium phosphate (K_2HPO_4), 0.2 magnesium sulfate ($MgSO_4 \cdot 7H_2O$), a trace amount of ferric sulfate ($Fe_2(SO_4)_3$), and 1000.0 ml of distilled water. After a preliminary heating of the medium, adjust pH to 6.8 with 3 *N* HCl or 3 *N* NaOH. Sterilize medium by autoclaving for 30 minutes at 15 psi.

4. Yeast-extract agar (Stevenson and Rovatt, 1953): Mix 15.0 g agar, 1.0 g dextrose, 1.0 g sodium chloride (NaCl), 0.01 g ferric chloride ($FeCl_3$), 1.0 g yeast extract, and 1000.0 ml distilled water. Adjust pH to 6.8 with 3 *N* HCl or 3 *N* NaOH. Sterilize medium by autoclaving for 30 minutes at 15 psi.

3.5.3.4 Materials--

1. Bottle, French square, 237 ml (8 oz) with caps. NOTE: 8 required per sample.

2. Three dozen spherical glass beads of 2 mm (0.079 in) diameter.

3. Autoclave, steam, capable of holding 15 psi and 121°C.

4. Sieve, 2 mm (10 mesh) openings.

5. Shaker, horizontal reciprocating type, 6.3 cm (2.5 in) stroke, 120 strokes per minute.

6. 10 ml pipette, sterile

7. 1 ml pipette, sterile.

8. Petri dishes, sterile. NOTE: 15 required per sample.

9. Balance, can be read to 0.01 g.

10. Humidified incubator. NOTE: A glass container with moistened paper towels in the bottom can be used. Place container in an oven.

11. Quebec colony counter or a wide-field, low-power microscope.

12. Sample bags.

3.5.3.5 Procedure (Adapted from Clark, 1965)--

1. From a thoroughly mixed bulk sample, transfer about 900 g of soil to a polyethylene bag for transport to the laboratory. NOTE: Containers must be clean and at least sanitized so as not to harbor other microorganisms not in the bulk sample. Avoid exposing the sample to heat or drying. If the sample is not used that day, it may be stored in a closed container (pinholed for aeration) at 4°C for 1 or 2 weeks without serious detriment.

2. Pass entire sample through a 2 mm sieve.

3. Mix sample thoroughly.

4. Withdraw a 10 g subsample and weigh. Record weight (A). Oven dry subsample in weighing container, cool in desiccator, and reweigh. Record weight (B). Determine soil moisture.

5. Put approximately 3 dozen spherical glass beads and 95 ml of water in a 237 ml screw cap bottle. NOTE: The purpose of the beads is to facilitate disintegration of soil aggregates.

6. To seven 237 ml screw cap bottles add 90 ml of water and no beads. NOTE: More than 7 bottles will be required for dilution series if sample is high in microorganisms. Less than 7 bottles required if low in microorganisms.

7. Cap all bottles.

8. Sterilize bottles by autoclaving at 15 pounds pressure for 15 minutes and cool to room temperature prior to use. Make sure caps are loose during autoclaving.

9. Transfer 10 g of moist soil into the bottle containing 95 ml of water and glass beads.

10. Tightly cap bottle.

11. Shake bottle containing sample for 3 minutes in a horizontal position in a reciprocating shaker or for an equal time by hand.

12. No longer than 10 minutes after removing the bottle from the shaker, shake bottle vigorously by hand for a few seconds and immediately transfer 10 ml from the center of the suspension to a bottle containing 90 ml water and no beads, using a sterile 10 ml pipette. NOTE: This establishes a 10^{-2} dilution.

13. Continue this dilution process by similarly transferring 10 ml quantities to successive bottles of 90 ml and no beads to provide a dilution

series through 10^{-7}. NOTE: Experience with different soils will provide a basis for estimating whether the highest dilution will need to be no more than 10^{-6} or 10^{-7}, or whether it will need to be as high as 10^{-8} or 10^{-9}.

14. From the highest dilution prepared, transfer a 1 ml portion of the freshly agitated suspension to each of 5 sterile petri dishes by means of a sterile, 1 ml pipette. Shake a few times before withdrawing the 1 ml portion.

15. Make similar transfers from the two next lower dilutions into other dishes. Shake as above.

16. Into each petri dish, pour about 15 ml of soil-extract agar, which previously has been steamed sufficiently to insure complete melting, and then cooled to 42°C. NOTE: Agar media that are starting to solidify, at about 40°C, are not suitable for pouring into plates.

17. Immediately after adding agar, cover dish and carefully rotate by hand to swirl the agar and to insure its thorough mixing with the inoculant. CAUTION: Do not splash medium-sample mixture on petri dish cover. If this should occur, discard and replace.

18. Permit poured plates to stand upright until the agar has solidified.

19. Invert plates in a humidified incubator at 28°C. NOTE: Some workers prefer 25°C; others 30°C. Do not use 37°C as is commonly the practice in medical bacteriology laboratories.

20. Leave the plates undisturbed for 4 days for fast growing bacteria. NOTE: Incubation time would depend upon type of bacteria being determined. If slow growing bacteria are being determined, 7 days are required and preferably 10 to 14 days. Actinomycetes require 10 days. Fungi can cover a medium if left more than 5 days. However, once a time period is established all samples must be counted at the established period of time.

21. Remove plates from the incubator.

22. Inspect all plates prepared from a single sample to see whether a proper dilution range has been plated and whether a proper dilution effect is apparent. NOTE: The proper dilution effect means that a plate prepared from a given dilution should have only approximately one-tenth as many colonies as the plate prepared from the next lower dilution. If there are numerous colonies on the plates or a dilution effect is not apparent, contamination has occurred. Discard all plates and rerun.

23. If incubation plates appear satisfactory, select the plates from the dilution at which 30 to 300 colonies have developed per plate. NOTE: (1) If the plate from the highest dilution shows greater than 300 colonies, the dilution has been too low. (2) If the lowest dilution shows less than 30 colonies, the dilution has been too high. In either event, discard all the plates. (3) If one or two plates within the 30 to 300 colony range have one or more large bacterial or fungal colonies (greater than 2 cm in diameter),

discard such plates without counting.

24. With the aid of a Quebec colony counter or a wide-field, low-power microscope, count the total number of colonies on each of the three or more remaining suitable plates.

3.5.3.6 Calculations--

1. Legend:

A = Weight moist soil.

B = Weight oven-dry soil.

2. Water loss = A - B.

3. Percent soil moisture = (Water loss/B) X 100.

4. Total viable count per gram of the initial moist soil sample = (average number of colonies per plate for a given dilution) X (dilution factor).

5. Grams of dry matter per gram of moist soil = B/A.

6. Total count per gram of dry soil = (count per gram of moist soil)/(grams of dry matter per gram of moist soil).

3.5.4 MPN of Aerobic Cellulose-Decomposing Bacteria

3.5.4.1 Principle--

The most-probable-number (MPN) method permits estimation of aerobic cellulose-decomposing bacteria without actually counting single cells or colonies. The method is based on the presence or absence of cellulose-decomposing bacteria on strips of paper. A strip of paper is needed for each dilution of a minesoil. A positive (or presence) reading indicates that at least one (it could be several) cellulose-decomposing bacterium was present (Alexander, 1965).

3.5.4.2 Comments--

Cellulose-decomposing bacteria must meet one of the following conditions: (1) Bacteria must bring about a change in the medium that is easily recognizable or (2) after the bacteria have multiplied, they must be recognizable on the strip of paper on which they are growing.

Single cellulose-decomposing bacterial cells must be capable of growth in the medium or the method is not reliable. That is, no growth in the medium without cellulose source, but growth with cellulose source added.

Some quantitative changes in the original number of bacteria can occur over a period of time, even with refrigeration. The samples should be passed through a 2.0 mm sieve to remove rocks and coal. Samples must be prepared the same day as collected (Alexander, 1965).

3.5.4.3 Chemicals--

NOTE: All chemicals must be ACS Certified pure grade.

1. Ammonium sulfate-cellulose solution (Fred and Waksman, 1928): Mix 1.0 g ammonium sulfate ($(NH_4)_2SO_4$), 1.0 g dipotassium phosphate (K_2HPO_4), 0.5 g magnesium sulfate ($MgSO_4 \cdot 7H_2O$), 2.0 g calcium carbonate ($CaCO_3$), trace amount of sodium chloride (NaCl), and 1000.0 ml of distilled water. NOTE: The $CaCO_3$ can be left out and a trace of $FeSO_4$ introduced.

3.5.4.4 Materials--

1. Samples sieved through 2 mm sieve from depth of 0-13 cm (0-5 in) or any other depth range of interest.

2. Medium-sized test tubes, 150 X 18 mm (6 X 0.7 in).

3. Strips of filter paper (see 3.5.4.5, No. 1).

4. Pipette, 1 ml, sterilized.

5. Microscope slides, glass, 7.62 X 2.54 cm (3 X 1 in), sterile.

6. Microscope with 10X or 15X eyepiece and 97X oil immersion objective.

7. Autoclave, steam, capable of holding 15 psi and 121°C.

8. Rubber stoppers (to fit test tubes).

3.5.4.5 Procedure (Fred and Waksman, 1928)--

1. Prepare a series of medium-sized test tubes containing 5 ml of the medium and a strip of filter paper. Part of the paper should protrude above the surface of the medium.

2. Plug test tubes with rubber stoppers. Sterilize by autoclaving for 15 minutes at 15 psi. NOTE: Make certain stoppers are loose or they will blow out. Fold a bit of paper and insert between tube and stopper before autoclaving and remove after autoclaving.

3. Prepare a 10-fold soil:water dilution series, stopping at 10^{-9}. (See 3.5.3.5 Steps 5 through 13).

4. Withdraw by sterile 1 ml pipette, 5 aliquots from the 10^{-9} soil suspension. Discharge 1 ml into each of 5 test tubes containing the medium.

5. Repeat step 4 for the next four lower dilutions, 10^{-8} through 10^{-5}. NOTE: Use lower dilutions if the number of organisms is expected to be small.

6. Incubate tubes at 25°C or 30°C.

7. Examine tubes daily.

8. Presence of cellulose-decomposing bacteria will be shown by the decomposition of the paper just at the surface of the liquid.

9. After 4 weeks storage, make final observations.

10. Record the number of tubes at each dilution in which growth has occurred as positive tubes.

11. Calculate the most-probable-number (MPN) of bacteria.

3.5.4.6 Calculations (Alexander, 1965)--

1. Select as P(1) the number of positive tubes of the least concentrated dilution in which all tubes are positive or in which the greatest number of tubes are positive.

2. P(2) and P(3) are the next two higher dilutions.

3. Using Table 13, find the row of numbers in which P(1) and P(2) correspond to the experimentally observed values.

4. Follow the row of numbers across the table to the column headed by the observed value of P(3). NOTE: This number is the MPN of organisms in the quantity of the original sample represented in the inoculum added in the second dilution, P(2) dilution factor.

5. Multiply the number found in step 4 by the dilution factor of P(2) to obtain the MPN for the original sample.

Example A -

Using a 10-fold dilution and 5 tubes per dilution, the following numbers of positive tubes were observed: 5 at 10^{-5}; 5 at 10^{-6}; 4 at 10^{-7}; 2 at 10^{-8}; 1 at 10^{-9}. In this series, P(1) = 5, P(2) = 4, and P(3) = 2. Table 13 gives a value of 2.2 for a dilution series of 10^{-7}, the dilution of P(2). Multiplying 2.2 times 10^7 gives a MPN for the original sample of 22 million bacteria, $2.2 \times 10^7 = 22,000,000$.

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6. The 95% confidence limits for MPN values can be determined from Table 14. Upper confidence limit at 95% level = (MPN value) X (factor from Table 14).

Lower confidence limit at 95% level = (MPN value)/(factor from Table 14).

TABLE 13. MOST-PROBABLE-NUMBERS FOR USE WITH 10-FOLD DILUTIONS AND 5 TUBES PER DILUTION (FROM COCHRAN, 1950)

P_1	P_2	Most probable number for indicated values of P_3					
		0	1	2	3	4	5
0	0	-	0.018	0.036	0.054	0.072	0.090
0	1	0.018	0.036	0.055	0.073	0.091	0.11
0	2	0.037	0.055	0.074	0.092	0.11	0.13
0	3	0.056	0.074	0.093	0.11	0.13	0.15
0	4	0.075	0.094	0.11	0.13	0.15	0.17
0	5	0.094	0.11	0.13	0.15	0.17	0.19
1	0	0.020	0.040	0.060	0.080	0.10	0.12
1	1	0.040	0.061	0.081	0.10	0.12	0.14
1	2	0.061	0.082	0.10	0.12	0.15	0.17
1	3	0.083	0.10	0.13	0.15	0.17	0.19
1	4	0.11	0.13	0.15	0.17	0.19	0.22
1	5	0.13	0.15	0.17	0.19	0.22	0.24
2	0	0.045	0.068	0.091	0.12	0.14	0.16
2	1	0.068	0.092	0.12	0.14	0.17	0.19
2	2	0.093	0.12	0.14	0.17	0.19	0.22
2	3	0.12	0.14	0.17	0.20	0.22	0.25
2	4	0.15	0.17	0.20	0.23	0.25	0.28
2	5	0.17	0.20	0.23	0.26	0.29	0.32
3	0	0.078	0.11	0.13	0.16	0.20	0.23
3	1	0.11	0.14	0.17	0.20	0.23	0.27
3	2	0.14	0.17	0.20	0.24	0.27	0.31
3	3	0.17	0.21	0.24	0.28	0.31	0.35
3	4	0.21	0.24	0.28	0.32	0.36	0.40
3	5	0.25	0.29	0.32	0.37	0.41	0.45
4	0	0.13	0.17	0.21	0.25	0.30	0.36
4	1	0.17	0.21	0.26	0.31	0.36	0.42
4	2	0.22	0.26	0.32	0.38	0.44	0.50
4	3	0.27	0.33	0.39	0.45	0.52	0.59
4	4	0.34	0.40	0.47	0.54	0.62	0.69
4	5	0.41	0.48	0.56	0.64	0.72	0.81
5	0	0.23	0.31	0.43	0.58	0.76	0.95
5	1	0.33	0.46	0.64	0.84	1.1	1.3
5	2	0.49	0.70	0.95	1.2	1.5	1.8
5	3	0.79	1.1	1.4	1.8	2.1	2.5
5	4	1.3	1.7	2.2	2.8	3.5	4.3
5	5	2.4	3.5	5.4	9.2	16	-

Example B -

The factor for example A using five tubes with a dilution of 10-fold and a MPN equaling 2.2 obtained from Table 14 is 3.30.

Upper confidence limit at 95% level = (2.2) X (3.30).

Upper confidence limit at 95% level = 7.26.

Lower confidence limit at 95% level = (2.2)/(3.3).

Lower confidence limit at 95% level = 0.67.

NOTE: New tables must be used if these particular number of tubes and dilutions are not used. MPN has a low order of precision. Large numbers of tubes must be inoculated for each dilution for precise estimates. Increasing the number of tubes inoculated at each dilution or narrowing the dilution ratio, reduces the confidence limit intervals at the 95% level.

3.5.5 Carbon Dioxide Production

3.5.5.1 Principle--

This method determines the amount of carbon dioxide produced, under laboratory conditions, by microbial decomposition of finely ground (40 mesh) straw (or any other additive). The quantity of carbon dioxide produced is an index of intensity for microbial activity. Minesoils, like other soils, have a microbial population. Vegetated minesoils are expected to contain larger numbers and a wider variety of microorganisms than nonvegetated minesoils (Hedrick and Wilson, 1956; Wilson and Hedrick, 1957b).

3.5.5.2 Comments--

The simplicity of this method is the ready accessibility of the materials. Care must be taken in preparation and standardization of the barium hydroxide, $Ba(OH)_2$, since exact concentration (Normality) is important (Hedrick and Wilson, 1956; Wilson and Hedrick, 1957b).

3.5.5.3 Chemicals--

NOTE: All chemicals must be ACS Certified pure grade.

1. Calcium hydroxide ($Ca(OH)_2$), 0.04 N, saturated solution: Dissolve 1.5 g (use some excess) of $Ca(OH)_2$ in carbon dioxide-free water (See 3.2.3.3 No. 1) and dilute to 1 liter. Filter off $CaCO_3$ and protect from CO_2 of the air with soda lime or ascarite in a guard tube.

2. Sodium nitrate ($NaNO_3$).

3. Calcium phosphate ($CaHPO_4$).

TABLE 14. FACTORS FOR CALCULATING THE CONFIDENCE LIMITS FOR THE MOST-PROBABLE-NUMBER COUNT (FROM COCHRAN, 1950)

No. of tubes per dilution (n)	Factor for 95% confidence limits with indicated dilution ratios			
	2	4	5	10
1	4.00	7.14	8.32	14.45
2	2.67	4.00	4.47	6.61
3	2.23	3.10	3.39	4.68
4	2.00	2.68	2.88	3.80
5	1.86	2.41	2.58	3.30
6	1.76	2.23	2.38	2.98
7	1.69	2.10	2.23	2.74
8	1.64	2.00	2.12	2.57
9	1.58	1.92	2.02	2.43
10	1.55	1.86	1.95	2.32

4. Monopotassium phosphate (KH_2PO_4).

5. Barium hydroxide ($Ba(OH)_2$), 0.1 N: Dissolve 15.75 g of $Ba(OH)_2$ in carbon dioxide-free water (See 3.2.3.3 No. 1) and dilute to 1 liter. Filter off $BaCO_3$ and protect from CO_2 of the air with soda lime or ascarite in a guard tube.

6. Hydrochloric acid (HCl), 0.1 N.

7. Phenolphthalein indicator.

3.5.5.4 Materials--

1. Sieve, 2 mm openings (10 mesh).

2. pH meter (Corning model 12 or equivalent) with combination electrode.

3. Refrigeration unit.

4. Straw, ground, 40 mesh, sterilized, or other biodegradable material.

5. Griffin beaker, 50 ml.

6. Griffin beaker, 600 ml.

7. Mason jars, 473.2 ml (1 pt).

8. Plastic cylinder, 3.81 cm (1.5 in) length.

9. Rubber stoppers.

10. Fiber board (pokerchip).

11. Metal lids, 2 piece to fit Mason jar.

12. Flasks, Erlenmeyer, 250 ml.

13. Balance, can be read to 0.1 g.

3.5.5.5 Procedure (Adapted from Hedrick and Wilson, 1956; Wilson and Hedrick, 1957b)--

1. Collect bulk samples representing the minesoil and depth in question, usually 0-8 cm (0-3 in).

2. Save material that will crush easily with fingers and pass through a 2 mm sieve.

3. Determine water-holding capacity (See 3.5.2.5, Steps 3 through 15).

4. Place 10.0 g of minesoil sample into a series of 250 ml Erlenmeyer flasks.

5. Add different amounts of 0.04 N $Ca(OH)_2$ to the flasks. NOTE: 5 ml of 0.04 N $Ca(OH)_2$ is equivalent of 1 ton of pulverized limestone per acre.

6. Dilute to 100 ml with distilled water.

7. Add 3 drops of chloroform. NOTE: The chloroform is added to prevent microbial activity.

8. Stopper flasks.

9. Thoroughly shake flasks twice a day.

10. Repeat step 9 for 4 days.

11. Determine pH values of the suspension.

12. Note the amount of 0.04 N $Ca(OH)_2$ required for 10.0 g of minesoil to have a pH of about 7.0.

13. Place 100 g of minesoil into each of two 600 ml beakers.
NOTE: One beaker will have the untreated sample and the other beaker will have the treated sample.

14. Adjust minesoil in both beakers to a pH of about 7.0 with $Ca(OH)_2$ using the data acquired from step 12.

15. To one beaker, add 1.0 g of ground straw, or other additive, and thoroughly mix.

16. To the same beaker add nitrogen (as $NaNO_3$), phosphorus (as $CaHPO_4$), and potassium (as KH_2PO_4) at an equivalent rate of 1000 lbs per acre of 4-12-4 fertilizer and thoroughly mix.

17. Close both ends of two 3.81 cm (1.5 in) plastic cylinders with rubber stoppers.

18. On one end cement a small disc of fiber board (pokerchip) to each cylinder.

19. Place a cylinder inside each of the Mason jars (incubation chambers) with the pokerchip end up.

20. While holding the cylinder firmly against the bottom, transfer the 100 g sample from the 600 ml beakers.

21. Bring minesoil to 50 percent water-holding capacity by the addition of distilled and/or deionized water.

22. Shake the jar gently to level the material and then gently tap it on a table to settle the material around the cylinder.

23. Place a 50 ml beaker containing 20 ml of 0.1 N $Ba(OH)_2$ and 7 drops of phenolphthalein on top of the pokerchip. NOTE: $Ba(OH)_2$ is used to absorb the CO_2 and the phenolphthalein is used as an indicator to show if the $Ba(OH)_2$ was converted to $BaCO_3$ before the one day incubation period was completed. If this occurs, quickly open the incubation chamber and replace with a new beaker of $Ba(OH)_2$ and note for the calculations.

24. Close the jars with two-piece metal lids.

25. After 24 hours, remove the 50 ml beakers from the Mason jars.

26. Titrate the $Ba(OH)_2$ with 0.1 N HCl until it clears.

27. Make a blank for each titration. NOTE: This is necessary to determine the amount of CO_2 in the stock $Ba(OH)_2$ solution.

28. After each titration, thoroughly aerate the incubation chamber by rapidly drawing carbon dioxide-free air into the jar for about 3 minutes.

29. Repeat steps 23 through 28 for 10 days.

3.5.5.6 Calculations--

1. ml $BaCO_3$ = (ml $Ba(OH)_2$ used) - (ml of HCl/2).

2. mg CO_2 = (ml $BaCO_3$) X (N of $Ba(OH)_2$ X 44).

3. Total mg CO_2/10 days = Sum total of mg CO_2 for each of the ten days.

3.5.6 MPN of Sulfur-Oxidizing Bacteria

3.5.6.1 Principle--

When sulfur is added to a minesoil, the sulfur at first oxidizes slowly. As the soil becomes acid, sulfur begins to oxidize rapidly.

Inoculation is made in a medium free of any organic compounds and carbonates. Sulfur is added as the only energy source. The bacteria convert sulfur into sulfuric acid thus lowering the pH.

3.5.6.2 Comments--

Since many organisms will not live in acid conditions, the medium has a reaction at about pH 4.0. The sulfur-oxidizing bacteria can develop at this low pH. The high acidity and high dilutions of the culture results in a pure culture.

Sterilization of the medium must be by flowing steam. The sterilization must be on 3 CONSECUTIVE days at 30 minutes each. This process is called intermittent sterilization. The first day kills vegetated cells; the second day kills spores that have germinated; and the third day kills any remaining vegetated cells. NOTE: Passing steam around the medium is the best procedure; however, an autoclave can be used if: (1) there is NO PRESSURE BUILDUP, and (2) temperature REMAINS at about 100°C.

The medium becomes turbid as bacterial growth develops and sulfur crystals can be seen in the medium. The medium also allows for pH determination.

3.5.6.3 Chemicals--

NOTE: All chemicals must be ACS Certified pure grade.

1. Sulfur-phosphate medium (Fred and Waksman, 1928): Mix 0.2 g ammonium sulfate ($(NH_4)_2SO_4$), 3.0 g monopotassium phosphate (KH_2PO_4), 0.25 g magnesium sulfate ($MgSO_4 \cdot 7H_2O$), a trace amount of ferrous sulfate ($FeSO_4 \cdot 7H_2O$), 10.0 g of powdered sulfur, and 1000.0 ml of distilled water. Weigh 1.0 g of sulfur into individual 250 ml Erlenmeyer flasks. Add 100 ml of the liquid medium to each flask. Reaction of the medium is about pH 4.0. Sterilize flasks in flowing steam for 30 minutes on 3 CONSECUTIVE days (See 3.5.6.2).

3.5.5.4 Materials--

1. Flasks, Erlenmeyer, 250 ml.

2. pH meter (Corning model 12 or equivalent) with combination electrode.

3. Microscope with 10X or 15X eyepiece.

4. Sieve, 2 mm (10 mesh) openings.

3.6.6.5 Procedure (Fred and Waksman, 1928)--

1. Sieve sample with 2 mm sieve.

2. Prepare 20 flasks with 100 ml of the medium.

3. Prepare a 10-fold soil:water dilution series, stopping at 10^{-9} (See 3.5.3.5 steps 5 through 13).

4. Withdraw by sterile 1 ml pipet, 5 aliquots from the 10^{-9} soil suspension. Discharge into flasks containing medium.

5. Repeat step 4 for the next four lower dilutions, 10^{-8} through 10^{-5}. NOTE: Use lower dilutions if the number of organisms is expected to be small.

6. Incubate flasks at 25° to 30°C.

7. After 7, 14, and 30 days, determine pH of flasks and note if medium has become turbid. NOTE: It is a good practice to check the turbid medium microscopically to determine if the turbidity is due to the presence of sulfur-oxidizing bacteria.

8. Record the number of flasks at each dilution in which turbidity has been observed. Record these tubes as positive tubes.

9. Determine the most-probable-number (MPN) of bacteria (See 3.5.4.6).

SECTION 4

SHORT TERM AND SIMULATED WEATHERING

4.1 LABORATORY AND FIELD METHODS

4.1.1 Summary

Physical and chemical changes inevitably occur in the changed environment of disturbed materials. The methods presented here provide a basis for estimating rate and degree of change.

A mild slaking test identifies materials that will disintegrate quickly when left exposed at the surface of a minesoil. These materials will provide fines in minesoil profiles.

The Physical Weathering Potential (P.W.P.) method disintegrates earth and rock fragments unless they are strongly cemented. Materials surviving this test should persist in loose rock flumes or valley fills. Fines measured by this method will form under intense weathering at the surface but will not necessarily form when rocks are covered in minesoils.

The modified Sieve Analysis after Intermediate Disaggregation (SAID) method disaggregates rock fragments less violently than P.W.P. but more than Standardized Slaking. Modified SAID provides an estimate of rock particles that soil scientists commonly consider coarse fragments rather than soil fines.

Field Weathering Plots evaluate rock stability or breakdown under exposed outdoor conditions in a particular climate. This method standardizes what happens on the surface of minesoils and helps to calibrate laboratory measurements of disintegration. It can be interpreted directly into recommended placement of disturbed materials and aids the study of variables that cause rock stability.

Stimulated Weathering Cells provide standard laboratory conditions for measuring rate and degree of change under favorable conditions for special reactions. The major focus has been sulfate and acid formation from pyritic forms of sulfur. The same approach applies to other chemical or physical changes.

4.1.2 Standardized Slaking

4.1.2.1 Principle--

An air-dry fragment of soil or rock when quickly submerged in water is subjected to forces that will break it apart if the individual grains are not firmly cemented together. The disruptive forces are caused by the release of air trapped in the pores of the fragment. As the water moves into the pore system, the air is compressed by surface tension causing pressures that may become great enough to break the fragment into small pieces.

4.1.2.2 Comments--

This method uses a mild treatment to get an index of physical weathering. It is simple, semiquantitative, and can be done in the field as well as the laboratory.

Many samples break into smaller pieces, but the pieces sometimes slump into a pile and do not fall through the 6.35 mm sieve. Physical overlap and surface tension may hold the small pieces of sample together on top of the sieve. To overcome this problem the standard liquid limit device has been tried and calibrated. It shakes the pieces apart and allows them to fall through the sieve.

4.1.2.3 Chemicals--

Distilled or tap water

4.1.2.4 Materials--

1. 250 ml beakers.

2. Hardware cloth, 6.35 mm (0.25 in) openings.

3. Paper clips.

4. Standard liquid limit device (Sowers, 1965, Fig. 1-1, p. 395) adjusted to drop a distance of 1 cm.

4.1.2.5 Procedure (Modified and updated from Smith et al., 1976)--

1. Select one or more rock fragments weighing approximately 15.0 g.

2. Cut hardware cloth to fit inside of beaker

3. Suspend the hardware cloth in the beaker by large paper clips hooked over the rim.

4. Fill beaker with enough water (distilled or tap) to cover sieve and fragment to be tested.

5. Place fragment on sieve and let sample stand undisturbed for 30 minutes.

6. Place beaker in cup of standard liquid limit device and turn crank 20 times at a rate of one revolution per second. NOTE: After every five revolutions, straighten beaker without changing the rate if necessary to keep the beaker upright.

7. Visually estimate the percentage of material which has fallen through the sieve, using a scale of 0 through 10 to represent 0 to 100 percent.

4.1.3 Physical Weathering Potential

4.1.3.1 Principle--

The procedure was developed by combining features of methods by Bouyoucos (1951), Tyner (1940), Day (1956), and Kilmer and Alexander (1949). Rocks are artificially weathered by treatment with a dispersing agent while shaking on a reciprocating shaker for 16 hours. The particle size distribution of material passing a 2 mm sieve is determined by mechanical analysis. In this procedure, materials are subjected to vigorous treatment to get a measure of particle sizes.

4.1.3.2 Comments--

The particle size distribution can be determined by either the hydrometer or pipet method. If the pipet method is used, be sure to read method 3.4.2 carefully before starting.

Temperature is quite important to the sedimentation procedure. Although correction factors are given, the procedure is best carried out in a constant temperature room or by placing the cylinders in a constant temperature bath. Care must be taken not to touch materials retained on the sieves with anything but a gentle stream of water during the washing process.

If the temperature corrected hydrometer reading for a particular size fraction equals the temperature corrected reading for the dispersing agent, that particle size is recorded as a "trace."

4.1.3.3 Chemicals--

Dispersing agent: Instant Calgon (see 3.2.2) or dissolve 35.7 g glassy sodium metaphosphate ($Na(PO_3)_6$) (Fisher Scientific No. S-333 or equivalent) and 7.94 g sodium carbonate (Na_2CO_3) in distilled water and dilute to one liter. The Na_2CO_3 is used as an alkaline buffer to prevent the hydrolysis of the metaphosphate back to the orthophosphate which occurs in acidic solutions.

4.1.3.4 Materials--

1. Bottles, French square, 1 liter (32 oz) with caps.

2. Shaker, horizontal reciprocating type, 6.3 cm (2.5 in) stroke, 120 strokes per minute.

3. Standard soil hydrometer (ASTM 152H, with Bouyoucos scale in grams per liter).

4. Glass sedimentation cylinders with markings at 1130 and 1205 ml levels (Bouyoucos cylinders).

5. Balance, can be read to 0.1 g.

6. Drying oven.

7. Weighing pans.

8. Thermometer, 0-100°F.

9. Plunger (see 3.4.3.4).

10. Sieve, 2 mm (10 mesh) openings, 13 cm (5 in) diameter.

11. Sieve, 6.35 mm (0.25 in) openings, 13 cm (5 in) diameter.

12. Powder funnel, large diameter to hold 2 mm sieve.

4.1.3.5 Procedure--

1. Dry intact rock fragments, between 13 and 38 mm in diameter, overnight in an oven set at 50°C.

2. The rock fragments are weighed (± 0.1 g) on a tared pan. Record as oven-dry weight (A). NOTE: Place no more than a 100.0 g sample of sandstone (no more than a 50.0 g sample of other rock types) in a one liter shaker bottle.

3. Add 125 ml of dispersing agent and 400 ml of distilled water to the bottle.

4. Cap bottle snugly and place horizontally on a reciprocating shaker for 16 hours at 120 strokes per minute.

5. Remove bottle and allow to cool to room temperature.

6. Put a wide-mouth powder funnel in a sedimentation cylinder and insert the 6.35 mm sieve on top of the 2 mm sieve in the funnel.

7. Transfer sample to sedimentation cylinder by pouring suspension through sieves. NOTE: Wash all sediment from bottle by holding bottle at a 45° angle with mouth of bottle over center of sieve. Direct a jet of distilled water upward into bottle, sweeping all particles out by the force of the water stream.

8. Carefully and thoroughly wash particles retained on sieves with a gentle stream of distilled water. CAUTION: Do not touch particles with anything but a stream of water. Do not exceed two-thirds the cylinder volume during washing.

9. Carefully remove sieves from funnel. Transfer material retained on each sieve to a separate tared weighing pan. NOTE: To make this transfer without losing material is important. The means of making the transfer can be with a jet of water, tapping material gently off sieves, picking material off by hand, etc.

10. Put weighing pan and material in oven at 105°C overnight. Weigh (± 0.1 g) material and record weight of greater than 6.35 mm material (B) and weight of 6.35 to 2 mm material (C).

11. Set cylinder in a place free from vibrations.

12. Place hydrometer in suspension.

13. Fill cylinder to upper mark (1205 ml) with distilled water for a sample between 50.0 and 100.0 g. Fill to lower mark (1130 ml) for 50.0 g sample.

14. Remove hydrometer. Take plunger in one hand holding cylinder with the other. Strongly move plunger up and down being careful not to spill contents of cylinder.

15. After all sediment is off the cylinder bottom, carefully remove plunger. Record time.

16. Record hydrometer reading at meniscus top at the end of 40 seconds. NOTE: About 10 seconds before taking reading, carefully insert hydrometer and steady by hand.

17. Remove hydrometer from suspension. CAUTION: Do not leave hydrometer in suspension longer than 20 seconds as particles will settle out on its shoulders.

18. Record suspension temperature. For each °F above calibrated temperature of the hydrometer, add 0.2 g to the reading. For each °F below calibrated temperature, subtract 0.2 g.

19. Record corrected hydrometer reading (D).

20. With the plunger, restir suspension. Take a reading at the end of 2 hours. Correct hydrometer reading (see step 18) and record corrected hydrometer reading (E).

21. Make 3 blanks by placing 125 ml of dispersing agent in 3 sedimentation cylinders.

22. Fill cylinder two-thirds full with distilled water. Insert hydrometer and fill cylinder to the lower mark (1130 ml) with distilled water.

23. Take hydrometer reading and temperature of suspension. Correct hydrometer reading using step 18 and record corrected hydrometer readings of the blanks (F_1, F_2, and F_3).

4.1.3.6 Calculations—

1. Legend:

A = Oven-dry wt. of rock fragments (excluding weighing pan).

B = Oven-dry wt. of material retained on 6.35 mm sieve (excluding weighing pan).

C = Oven-dry wt. of material retained on 2 mm sieve (excluding weighing pan).

D = Temperature corrected 40 second hydrometer reading.

E = Temperature corrected 2 hour hydrometer reading.

F_1 = Temperature corrected reading of first blank.

F_2 = Temperature corrected reading of second blank.

F_3 = Temperature corrected reading of third blank.

G = Dispersing agent correction factor.

2. $G = (F_1 + F_2 + F_3)/3$.

3. % material greater than 6.35 mm in diameter = (B/A) X 100.

4. % material between 2 and 6.35 mm in diameter = (C/A) X 100.

5. Weight corrected 2 hour reading = E - G.

6. Weight corrected 40 second reading = D - G.

7. % clay = (Weight corrected 2 hour reading/A) X 100.

8. % silt = [(Weight corrected 40 second reading - weight corrected 2 hour reading)/A] X 100.

9. % sand = 100 - (% material greater than 6.35 mm + % material between 2 and 6.35 mm + % clay + % silt).

4.1.4 Modified SAID

4.1.4.1 Principle--

This method uses a combination of slaking and a minimum of vigorous shaking to decompose a rock fragment. The suspension is passed through a nest of sieves and washed with water. The amount of material retained on each sieve is then calculated as a percent of the total sample.

4.1.4.2 Comments--

The term SAID was coined and defined by Soil Survey Staff (1970). The translation is: Sieve Analysis after Intermediate Disaggregation. Our modified SAID retains the original idea of the method but changes details to satisfy objectives of this manual.

The procedure is designed particularly for normal shales, mudstones, and other materials which tend to break-down easily. It provides a laboratory measurement needed to help predict field behavior and for correlation with other methods. It is intermediate in intensity between Slaking (4.1.2) and Physical Weathering Potential (4.1.3).

4.1.4.3 Chemicals--

1. Dispersing agent: Sodium metaphosphate ($Na(PO_3)_6$) available from Fisher Scientific Company No. S-333 or Instant Calgon available from Calgon Corporation, Pittsburgh, Pennsylvania).

2. Water, tap.

4.1.4.4 Materials--

1. Sieve, 6.35 mm (0.25 in) openings, U.S. Standard, 20.3 cm (8 in) diameter.

2. Sieve, 2 mm (10 mesh) openings, U.S. Standard, 20.3 cm (8 in) diameter.

3. Sieve, 0.1 mm (140 mesh) openings, U.S. Standard, 20.3 cm (8 in) diameter.

4. Balance, can be read 0.01 g.

5. Flasks, Erlenmeyer, 2 liter capacity with rubber stoppers.

6. Aluminum cake tins, 23 cm (9 in) diameter.

4.1.4.5 Procedure--

1. Rock fragments between 13 and 20 mm in diameter are air dried.

2. Take a representative sample of approximately 50 g. Weigh in tared pan and record weight (A).

3. Place sample in a 2 liter Erlenmeyer flask.

4. Add 1 liter of tap water and 1 heaping teaspoon (about 5 g) of dispersing agent.

5. Gently swirl, stopper, and let stand overnight.

6. Again swirl gently to free soil from bottom of flask.

7. Rotate end for end vigorously 10 times.

8. Pass through a nest of 3 sieves with 6.35 mm, 2.0 mm and 0.1 mm openings.

9. Wash the samples left on the sieves with a gentle stream of tap water and allow time for air drying.

10. Vigorously shake the air-dry separates from side to side on sieves for 1 minute.

11. Weigh material retained on 6.35 mm sieve in tared pan and record weight (B).

12. Weigh material retained on 2 mm sieve in tared pan and record weight (C).

13. Weigh material retained on 0.1 mm sieve in tared pan and record weight (D).

4.1.4.6 Calculations--

1. Legend:

A = Air-dry wt. of sample (excluding weighing pan).

B = Air-dry wt. of material retained on 6.35 mm sieve (excluding weighing pan).

C = Air-dry wt. of material retained on 2 mm sieve (excluding weighing pan).

D = Air-dry wt. of material retained on 0.1 mm sieve (excluding weighing pan).

2. % material greater than 6.35 mm = (B/A) X 100.

3. % material between 2 and 6.35 mm = (C/A) X 100.

4. % material between 0.1 and 2 mm = (D/A) X 100.

5. % material less than 6.35 mm = 100 - (calculation no. 2).

6. % material less than 0.1 mm = 100 - (calculation no. 2 + no. 3 + no. 4).

4.1.5 Simulated Weathering Cells

4.1.5.1 Principle —

Processes of chemical weathering that take place during acid generation are: solution, oxidation, hydration, and hydrolysis, with oxidation usually emphasized. Carrucio (1967) stated that the oxidation of pyrite was affected by four factors: oxygen, temperature, mode of iron disulfide, and bacteria. He refined laboratory cells to provide standard conditions for measuring acid generation rates of selected materials. Other variables influencing rates include exposed surface area, catalytic agents, pH, ferric iron, and mineral species other than iron disulfide. The cells described here provide simple control over air, temperature, moisture, and microbes. Conditions created are relatively favorable to formation of sulfates and related compounds. Different materials can be compared and rated. Special treatments (i.e. lime rates) can be imposed to help answer theoretical and practical questions.

The end products have to be removed or the rate of oxidation will decrease. Decomposition products form coatings on particles and effectively close off the exposed surfaces. The decomposition products are removed by leaching the sample with distilled water at the end of each treatment cycle. Measurements are then made on the leachate.

4.1.5.2 Comments—

Empirical comparisons of materials and treatments afforded by this method may be interpreted directly into likely field behavior or may serve to reinforce or calibrate other laboratory measurements of field experiences. Analyses identified will give a good indication of major reactions occurring in samples. Additional analyses will satisfy specific objectives related to plant nutrients or toxic elements.

For each cell, the graduated cylinder, beaker, and centrifuge tubes into which the water extract is poured should be labeled the same as the cell. It is better to take a little time labelling everything clearly and distinctly, than to save time and get two or three samples intermixed.

4.1.5.3 Chemicals—

1. Distilled water (H_2O).

2. Sodium hydroxide (NaOH), 1 N stock solution : Dissolve 40.0 g NaOH (electrolytic pellets) in carbon dioxide-free, distilled water (see 3.2.3.3 No. 1) and dilute to 1 liter. Protect from the atmosphere using an ascarite guard tube.

3. Sodium hydroxide (NaOH), 0.01 N : Pipet 10 ml of 1 N NaOH into a 1 liter volumetric flask and dilute to volume with carbon dioxide-free, distilled water (see 3.2.3.3 No. 1). Protect from the atmosphere using an ascarite guard tube.

4.1.5.4 Materials--

1. Plastic shoe box. The plastic shoe box is used to make a leaching chamber. The chamber is constructed by drilling a 6.35 mm (0.25 in) hole in the center of one of the short sides of the box. Reverse box and drill a 6.35 mm (0.25 in) hole at the base of the other short side in the right hand corner. Plexiglass tubing is inserted and bonded with a nonwater-soluble glue to the box. Be sure that the plexiglass tubing bonded to the box is 2.5 cm (1 in) long with equal lengths on both the inside and outside of box. The hole in the center is used for the entrance of dry and moistened air. While the hole in the corner is used as an exit. The lid should be sealed using a non-hardening putty weatherstripping.

2. Wide-mouth jar, screw lid. (Mason type) - The jar is used as a humidifier. Two 6.35 mm (0.25 in) holes are drilled in the lid and copper tubing, 2.5 cm (1 in) long, is inserted and soldered in place. From the bottom of the lid flexible plastic tubing is attached to one piece of copper tubing and to an aerating stone, which rests on the bottom of the bottle. The copper tubing, which is attached to the aerating stone inside the bottle, is attached to an external air source. To the other copper tubing insert, attach flexible plastic tubing to the inlet tube of the leaching chamber.

3. Aerating stone.

4. Pipet, 20 ml volumetric.

5. Flexible plastic tubing (Tygon or equivalent).

6. Compressed air source.

7. Sieve, 2 mm (10 mesh) openings.

8. Graduated cylinders, 100 ml capacity, plastic or glass.

9. Microburet, 10 ml capacity, graduations in 0.02 ml (Kimax 17110F or equivalent).

10. Meniscus magnifier for above buret.

11. pH meter (Corning Model 12 or Equivalent), with combination electrode.

12. Wheatstone bridge, (see 3.2.18.4, no. 1).

13. HACH water test kit (Model DR-EL or equivalent).

14. Conductivity cell, pipet type, with cell constant of 1.0 reciprocal centimeter.

15. Beaker, 250 ml.

4.1.5.5 Procedure--

1. Crush sample to pass a 2 mm sieve. NOTE: Subsampling and grinding are done according to method 3.1.2; however, after the material has been crushed to 6.35 mm (0.25 in) size, the material is split into equal halves. One-half is used for this weathering experiment. The other half is subsampled and ground according to method 3.1.2 for chemical analyses.

2. Place 200 g of less than 2 mm material in the specially designed cell and spread evenly across the bottom. The sample is then thoroughly moistened with distilled water. NOTE: At this time the cell and sample can be inoculated with bacteria to catalyze the oxidation reaction.

3. The lid is sealed tightly to the bottom of the cell and the air line is attached to the air source. The experiment runs in a 7 day cycle. For the first three days, dry air is passed over the sample. Then for the next three days, moistened air is passed over the sample by filling the widemouth jar about half full with water and allowing air from the aerating stone to pass through the water.

4. On the last day of the cycle, 200 ml of distilled water is added to each cell. The sample is allowed to soak for one hour.

5. After soaking, the cell is drained through the plexiglass tubing at the front of the cell into a beaker. NOTE: If the water extract is turbid, spin the water extract down in a centrifuge. Pour off the supernatant liquid into a graduated cylinder and set aside. With a small amount of distilled water, resuspend the fine material in the bottom of the centrifuge tube and pour back into its proper cell.

6. The cycle of dry air and moist air passing over the sample in the cell is started over.

7. The water extract from each cell is measured in a graduated cylinder and the volume recorded (A).

8. Measure the electrical conductivity (see 3.2.18.5) of the water extract.

9. A 25 ml aliquot of the water extract will be taken and used to measure sulfate concentration using a HACH DR-EL kit. Record instrument reading of sulfate concentration (B).

10. Measure the pH (see 3.2.2.5) of the water extract. NOTE: When the pH of water extract is 7.0 or higher, titratable acidity is zero and steps 11 through 13 are omitted.

11. The remaining water extract is transferred to a beaker and heated to boiling to drive off any dissolved CO_2.

12. After the water extract has boiled for one minute the beaker is transferred to a desiccator, which contains no dessicant, and allowed to

come to room temperature. NOTE: An ascarite tube is placed inside the desiccator to remove any CO_2 in the air.

13. After cooling, the water extract is titrated to pH 7.0 with 0.01 N NaOH. Record volume of titrant used (C).

4.1.5.6 Calculations--

1. Legend:

A = Volume of water extract.

B = Sulfate concentration (ppm).

C = Volume of titrant used.

TS = Total sulfates (mg/100 g).

TA = Titratable acidity (ppm).

DF = Dilution factor.

2. Electrical conductivity. This measurement will be recorded for each water extract and will be plotted on a graph versus time.

3. pH. This measurement will be made on each water extract and will be plotted on a graph versus time.

4. $TS = [B \times DF \times (A/25)]/200$, where $DF = 25/\text{volume of extract used}$. This measurement will be made on each water extract. It will be plotted on a graph versus time. Also, it will be plotted as accumulative sulfates released versus time.

5. $TA = [(C \times 0.01\ \underline{N}) \times (A/(A - 25)) \times 5]$. Titratable acidity will be plotted on a graph versus time. On another graph, accumulative titratable acidity versus time will be plotted.

4.1.6 Field Weathering Plots

4.1.6.1 Principle--

A group of standard plots under uniform outdoor conditions constitutes a weathering yard for that particular climate. The purpose of a weathering yard is to provide a standard near-natural means of comparing and rating the stability of rock or earth fragments selected to represent materials of special interest. Standard exposure affords comparison between paired samples and calibration of breakdown against descriptive properties, laboratory measurements, and weather events. Periodic descriptions, photographs, and weights record sample changes over time. Special tests involve surface placement versus shallow burial, flat versus upright orientation of bedded samples, contact with acid versus alkaline substrate, and other variables. This yard provides ratings that can help operators and regulatory people decide which rocks to choose for selective placement for a particular purpose.

4.1.6.2 Comments--

Care must be exercised to insure that representative samples of a rock are selected for exposure in the weathering yard. To facilitate observations and hamper sample manipulation by rodents, vegetative growth in the plot rows is controlled with periodic sprayings of herbicide.

Plastic fence should be periodically checked to insure their security, especially during periods of freezing and thawing of the ground. If the fence is not securely in the ground, the sand cushion may be lost. A sand cushion was chosen because rodents tended to remove cushions of nylon or cloth. A coarse (0.84 - 0.50 mm), pure silica sand is used to facilitate the separation of the weathered particles from the cushion. Due to its inert nature, pure silica will not markedly change size nor affect the sample weathering.

A grid system was utilized to facilitate sample location and number. The even numbered rows contain 25 plots each, designated "A" through "Y". The odd numbered rows allow passage through the weathering yard and contain no plots. Each plot is 2 X 2 m with space for four subplots designated "a" through "d" in a clockwise direction from the upper left corner. The weathering yard should be located away from possible flood or other damage and in an open area where samples will not be sheltered from sun or rain actions. Duplicate samples should be evaluated.

4.1.6.3 Chemicals--

1. Herbicide (Paraquot or equivalent).

2. Hydrochloric acid (HCl), 1 part acid to 3 parts water : Dilute 250 ml of concentrated HCl to 1 liter with distilled water.

4.1.6.4 Materials--

1. Plastic yard trimming fence, 15 cm (6 in) high, cut in 60 cm (24 in) lengths. A circle is formed from each length, overlapping the ends by about 5 cm (2 in) and stapling together with 6 staples.

2. Pure silica sand, 0.84 to 0.50 mm diameter (Ottawa flint shot, Berkeley Springs 2Z sand, or equivalent). The sand is sieved through a nested 20 mesh (0.84 mm openings) sieve and a 35 mesh (0.50 mm openings) sieve with a 2 minute hand shaking time. Sand retained on the 35 mesh sieve is used on the weathering plots.

3. Sieve, 0.84 mm (20 mesh) openings, 20.3 cm (8 in) diameter.

4. Sieve, 0.50 mm (35 mesh) openings, 20.3 cm (8 in) diameter.

5. Balance, can be read to 0.01 g.

6. Tile spade.

7. Metric ruler.

8. Munsell color book (available from Munsell Color Division, Kollmorgen Corporation, Baltimore, Maryland 21218).

9. Knife.

10. Weather station (optional). Required if accurate weathering conditions are required.

4.1.6.5 Procedure--

1. Select a 5 to 10 cm representative rock sample.

2. Weigh sample to the nearest 0.01 g.

3. Obtain a Munsell color of the matrix, streak, and any noticeable spots or films (see 2.1.3).

4. Determine rock type (see 2.1.2) and record hardness (see 2.1.4).

5. Check for presence of calcareous material (see 2.1.5), and other rock features (see 2.1.6 and 2.1.7).

6. Dig a round hole with the tile spade 15 cm (6 in) in diameter and 5 cm (2 in) deep.

7. Place prepared plastic fence in hole, placing soil around both the inside and the outside to hold it in place. Leave the inside depth 4 cm (1.5 in) below ground level.

8. Place 4 cm (1.5 in) of prepared pure silica sand inside the plastic fence.

9. Place rock sample on the sand.

10. Describe samples periodically in the field using the following criteria:

a. Is it breaking down? If so, describe.

b. If cracks are developing, are they regular or irregular?

c. Have there been any color changes?

d. Are there any other unusual qualities of the sample?

11. A daily record of high and low temperature and rainfall should be maintained.

12. Carefully take intact samples into laboratory periodically for weighing.

13. After samples have been broken down, remove them from the field, weigh and do a hydrometer mechanical analysis (see 3.4.4) on the less than 2 mm fractions.

14. At the end of a study remove the remaining resistant rocks, describe, weigh, and report their percent weight loss.

4.1.6.6 Calculations--

% weight loss = [(Initial wt. - Terminal wt.)/Initial wt.] X 100.

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GLOSSARY

Acidic - Having a pH of less than 7.0

Actinomycetes - Any of numerous generally filamentous and often pathogenic microorganisms of the family Actinomycetaceae, resembling both bacteria and fungi.

Aerobic - Oxygen-requiring organism.

Agar - Dried polysaccharide extract of red algae used as a solidifying agent in microbiological media.

Agricultural Limestone - A soil amendment consisting principally of calcium carbonate but including magnesium carbonate and perhaps other materials used to furnish calcium and magnesium as essential elements for the growth of plants and to neutralize soil acidity. It is made by crushing or pulverizing limestone.

Argillians - See 3.3.3.2.

Alkaline - Having a pH greater than 7.0.

Amorphous - Having no crystal structure.

Anistrophic (optical mineralogy) - Any crystal in which the optical properties (indexes) very with respect to directions of the crystal axes. Term includes all crystal systems except isometric and amorphous.

Anthropic - Said of an epipedon that is similar to a mollic epipedon but in which the content of P_2O_5 is greater than 250 ppm.

Argillaceous - Containing an appreciable amount of clay-size particles.

Autoclave - An apparatus using steam under pressure for sterilization.

Bacteria - Any of numerous unicellular microorganisms occurring in a wide variety of forms, existing as either a free-living organism or parasite, and having a wide range of biochemical, often pathogenic, properties.

Bedding Planes - Planes that mark the breaks between different rock types or changes in color or texture within a rock type.

Biotite - A mineral of the mica group. (See 3.3.2.2 for optical properties).

Birefringence - See 3.3.2.2.

Blossom - The decomposed (weathered) outcrop of a coal seam on the land surface.

Bone Coal - Coal in which the content of earthy material is too high to be commercially valuable; the percent of ash ranges upward from about 25 percent; bone is dull rather than bright, is both heavier and harder than commercial coal, and is classified for this manual as the rock type carbolith.

Canada balsam - a viscous, yellowish, transparent resin obtained from the balsam fir and used as a mounting cement for microscopic specimens.

Carbolith - See 2.1.2.

Carbonate - A group of minerals which all contain the anion $(CO_3)^{-2}$.

Chalcopyrite - A sulfide of copper and iron, $CuFeS_2$.

Chert - See 2.1.2.

Chlorite - A group of clay minerals as defined by method 3.3.4.

Clay film - See clay skin.

Clay-size particles - Particles having an equivalent diameter of less than 2 microns (0.002 mm).

Clay skin (clay film) - A thin coating of well-oriented clay particles on the surface of a soil aggregate, particle, or pore.

Collapsing minerals - A group of clay minerals whose internal structure collapses upon heating due to the removal of interlayer waters.

Colony - A microscopically visible growth of microorganisms on a solid culture medium.

Concretion - A hard, compact, rounded, normally subspherical mass or aggregate of mineral matter generally formed by orderly and localized precipitation from an aqueous solution in the pores of a sedimentary rock and usually of a composition widely different from that of a surrounding rock and rather sharply separated from it. Concretions have concentric layering about a central core.

Confidence limits - Either the upper or lower value between which an actual measurement or parameter will fall with a stated probability.

Crystal - A homogeneous, solid body of a chemical element, compound, or isomorphous mixture having a regularly repeating atomic arrangement that may be outwardly expressed by planar faces.

Culture - A population of microorganisms grown in a medium.

Cutans - See 3.3.3.2.

Density - The weight of a substance per unit volume of water which it will displace. Bulk density is expressed in grams per cubic centimeter.

Dolomite - Same as limestone except has substitution of magnesium for some of the calcium, CaMg $(CO_3)_2$. Differs from limestone since cold dilute HCl will not, or only slightly, cause effervesence except when applied to powdered sample.

Drift - See 1.1.2.

Earth worm casts - See 3.3.3.2.

Earthy material - See 2.1.2.

Epipedon - A diagnostic surface layer of soil.

Epsomite - See 2.1.7.

Extinction - The more or less complete darkness obtained in a birefringent mineral at two positions during a complete rotation as seen with crossed nicols using a petrographic microscope.

Fe-Al sulfates - See 2.1.7.

Ferrans - See 3.3.3.2.

Fizz - The process of a material (whether rock or soil) bubbling when cold dilute hydrochloric acid is applied.

Flint - See 2.1.2.

Fossils - Any remains, trace, or imprint of a plant or animal that has been preserved, by natural processes, in the earth's crust since some past geologic time.

Fungi - Any of numerous plants including the yeasts, molds, smuts, and mushrooms.

Glacial drift - See 2.1.2.

Gypsum - See 2.1.7.

Hardness - The resistance of a mineral to scratching (See 2.1.4 for more detail).

Hematite - An iron oxide (Fe_2O_3) mineral (See 3.3.2.2 for optical properties).

Hexagonal - One of the six crystal systems characterized by minerals having six crystal faces resulting from three equal length horizontal axes at right angles to a differing length central vertical axis.

Highwall - The exposed face of excavated overburden and coal in a surface mine or the face or bank of the uphill side of a contour strip mine excavation.

Horizon - See soil horizon.

Hydrated Lime - A material made from burnt lime which consists primarily of calcium hydroxide.

Illite - A group of clay minerals as defined by method 3.3.4.

Immersion - To cover completely with a liquid.

Incubate (incubation) - Holding cultures of microorganisms under conditions, especially temperature, favorable to their growth.

Incubation period - The time period during which microorganisms innoculated into a medium are allowed to grow.

Inoculum - The material containing microorganisms and used for the artificial introduction of microorganisms into a culture medium.

Intercalate - See 2.1.2.

Interference colors - See 3.3.2.2.

Isometric - One of the six crystal systems characterized by three axes that are mutually perpendicular and of equal lengths.

Isotrophic - See 3.3.2.2.

Kaolin - A group of clay minerals as defined by method 3.3.4.

Kaolinite - A clay mineral of the kaolin group as defined by method 3.3.4.

Jasper - See 2.1.2

Limestone - See 2.1.2.

Loess - See 2.1.2.

Luster - The reflection of light from the surface of a mineral.

Macroscopic - Visible without the aid of a microscope.

Mangans - See 3.3.3.2.

Marcasite - Like pyrite, an iron disulfide, FeS_2; however, differs in crystal form.

Medium - A substance used to provide nutrients for the growth and multiplication of microorganisms.

Mica - See 2.1.6.

Microcline - A mineral of the alkali feldspar group. (See 3.3.2.2 for optical properties).

Microbe (Microbial) - Microscopic organism belonging to either the plant or animal kingdom.

Microorganism - Form of life of microscopic dimensions.

Microscopic - Visible only with the aid of a microscope.

Mineral - A naturally formed chemical element or compound having a definite chemical composition and usually a characteristic crystal form.

Mollic - Pertaining to a dark, thick epipedon having at least 0.58% organic carbon, a base saturation of at least 50% when measured at pH 7, and less than 250 ppm P_2O_5 soluble in citric acid.

Monoclinic - One of the six crystal systems. Minerals in this system will have three unequal axes, two of which are obliquely inclined to each other and the third is perpendicular to the plane formed by them.

Montmorillonite - A group of expanding-lattice clay minerals as defined by method 3.3.4.

Mudrock - See 2.1.2.

Mudstone - See 2.1.2.

Munsell Color System - A color designation system that specifies the relative degrees of the three simple variables of color: hue, value, and, chroma. The Hue of a color indicates its relation to red, yellow, green, blue, or purple; the Value indicates its lightness (ranges from absolute black at 0 to absolute white at 10); The Chroma indicates its strength (or departure from neutral of the same lightness). For example: 10YR 6/4 has a color of Hue 10YR, Value 6, and Chroma 4 (for additional information see 2.1.3).

Muscovite - A mineral of the mica group (see 3.3.2.2 for properties).

Nodule - A hard, compact, rounded mass or aggregate of mineral matter. Unlike concretions, nodules do not have concentric layering.

Opaque - Said of materials that are impervious to light.

Organism - A living biological specimen.

Orthoclase - A mineral of the alkali feldspar group. (See 3.3.2.2 for properties).

Orthorhombic - One of the six crystal systems, characterized by three axes that are mutually perpendicular and of unequal length.

Outwash - See 2.1.2.

pH - A numberical measure of the acidity or alkalinity. The neutral point is pH 7.0. All pH values below 7.0 are acid and all above 7.0 are alkaline (for more information see 3.2.2).

Plate counting - The counting of microorganisms on a microscope slide.

Pocket - A small, discontinuous occurrence or patch of mineralized material, rock, soil, or void within a rock, stratum, or soil.

Polysaccharide - A carbohydrate formed by the combination of many molecules of monosaccharides (e.g. starch, cellulose, glycogen).

Pores - See 3.3.3.2.

Pyrite - An iron disulfide (FeS_2) mineral. (See 2.1.6 for detection and 3.3.2.2 for optical properties).

Quartz - Crystalline silica (SiO_2). (See 3.3.2.2 for optical properties).

Rock - Any consolidated or coherent naturally formed mass of mineral matter.

Rock chips - (a) Field - Fragments of rock expelled by a compressed air rotary blast hole drill.
(b) Laboratory - Fragments taken from a rock used for analysis.

Rock Texture - A general physical appearance or character of a rock and the mutual relations among the component particles or crystals; e.g. the size, shape, and arrangement of the constituent elements of a sedimentary rock.

Root channels - See 3.3.3.2.

Sand-size particles - A particle having an equivalent diameter in the range of 0.05 to 2.0 mm.

Sandstone - See 2.1.2.

Shale - See 2.1.2.

Silt-size particles - A particle having an equivalent diameter in the range of 0.002 to 0.05 mm.

Siltstone - A fine-grained consolidated clastic rock composed predominantly of particles of silt grade or size. Individual grains are not visible without magnification. Crushed wet fragments feel smooth rather than gritty or sticky. Would fall into the mudrock or mudstone category depending on hardness.

Skeletal grains - See 3.3.3.2.

Soil - The natural bodies on the earth's surface, in places modified or even made by man of earthy materials containing living matter and supporting or capable of supporting plants out-of-doors.

Soil horizon - A layer within the soil profile that has characteristics that separate it from the rest of the profile. (For additional information on the soil horizons used in the manual, see 2.1.2).

Soil profile - A vertical section of the soil from the surface through all its horizons.

Soil texture - The relative proportion of the various soil separates (e.g. sand, clay, and silt) in a soil. (see 2.1.8 for additional information).

Spore - A resistant body formed by certain microorganisms; a resistant resting cell; a primitive unicellular reproductive body.

Sterilize (Sterilization) - The killing of all forms of life.

Stokes' Law - A formula that expresses the rates of settling of spherical particles in a fluid: $V = Cr^2$, where V is velocity (in cm/sec), r is the particles radius (in cm), and C is a constant relating relative densities of fluid and particle, acceleration due to gravity, and viscosity of the fluid.

Tension - The suction or negative pressure of soil water.

Tetragonal - One of the six crystal systems, in which the crystals are related to three mutually perpendicular axes, the vertical axis of which is of unequal length relative to the two horizontal axes.

Thin section - See 3.3.3.1.

Translucent - Said of a mineral that transmits light, but is not transparent.

Transparent - Said of a mineral that is capable of transmitting light, and through which an object can be seen.

Triclinic - One of the six crystal systems, characterized by three unequal axes that intersect obliquely.

Texture - See rock texture or soil texture.

Till - See 2.1.2.

Umbric - Pertaining to an epipedon that is similar to a mollic epipedon except for having a base saturation of less than 50%, measured at a pH of 7.

Undersoil - The material (rock or unconsolidated) that directly underlies a coal.

Vermiculite - A group of clay minerals as defined by method 3.3.4.

Viable - Living

Viscosity - The property of a substance to offer internal resistance to flow.

Water holding capacity - The smallest value to which the water content of a soil can be reduced by gravity drainage.